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Considerations in the Selection of Ring-Spring Materials for the Quick Disconnect Vacuum Coupling

SUMMARY

During the evaluation of candidate materials for the ring-spring portion of the quick-disconnect vacuum coupling, several problems associated with the BSY environment were investigated. The materials were evaluated with regard to their response to: 1) general corrosion, 2) stress corrosion, and 3) hydrogen embrittlement. 17-4 PH stainless steel was selected as the ring-spring material. Also covered are discussions with the casting vendor and a short heat-treatment study to investigate modifying the 17-4 PH cast structure. Recommendations for a surveillance program are also made.

I. INTRODUCTION

A quick-disconnect vacuum coupling has been designed to facilitate the rapid insertion and removal of monitoring equipment and experimental apparatus in the Beam Switchyard (BSY). A critical portion of this assembly is the ring spring which distributes a uniform peripheral load to the assembly from compressed coiled springs. These coiled springs transmit their force to the ring springs through a connection near the ring-spring "ears". In service, the load from the compressed springs will be distributed from the ring ears to the assembly by means of eight (8) raised lugs positioned around the ring spring.

The choice for the ring-spring material, 17-4 PH stainless steel heat treated to an H900 condition, was reviewed in light of expected environmental conditions in the Beam Switchyard. Also considered were other possible material choices for the ring springs, alternate heat treatments, and the effects of manufacturing processes upon the material selected.

Included, therefore, in this report is a short discussion of the major problems which may cause premature ring-spring failure and their relationship to the selected material. The BSY environment, which is now only generally understood, limits the possible ring material choices available with a less stringent environment, so a wide range of potential problems possibly relating to other BSY components was included in the study. Briefly, the problems which were reviewed included general corrosion mechanisms, stress-corrosion cracking, and hydrogen embrittlement. At the end of the report, suggestions are given which may help to avoid catastrophic down time due to failures from an unexpected cause.

II. DISCUSSION

In order to properly evaluate choices for the ring-spring material, it was first necessary to define the BSY environment and the possible effects of this environment on various materials. Following a short discussion of these effects, the merits of several candidate materials follow.

Finally, a summary of discussions with the ring-spring casting vendor and the results of a short heat-treating study on the selected material is presented.

A. Beam Switchyard Environment

In addition to normal room temperature air containing a normal amount of moisture, the general BSY environment will include several chemicals produced by particle bombardment from the high-energy electron beam. It has been noted[1] that the first, and probably most prevalent, of these chemicals to be produced will be nitrous oxide which, in the presence of water, will form nitric acid. Other possible chemicals that may form will be oxides of sulfur and tellurium from dry lubricants and possibly HCl from residue films left on components after various cleaning and degreasing processes. All of these chemicals produce corrosive acids when combined with water.

Also to be considered as part of the BSY environment is the electron beam and its generated stream of particles. According to Neet[2] a fast neutron flux up to 10^{19} neutrons (2×10^{11} rads) can be expected. The effects on materials of construction will be to embrittle and strengthen, but after some period of time, element transmutation will begin to alter alloy structure. The net effect of these changes will be to generally degrade the material properties from their original design criteria.

B. Possible Environmental Effects

Of the many possible environmental effects that can change the role of constructural materials, three effects important to the BSY application will be considered here. These are general corrosion, stress corrosion, and hydrogen embrittlement.

1. General Corrosion Problems Related to the Stainless Steels

It was assumed that the most prevalent corrosion medium in the BSY would be nitric acid. Stainless steels were first used commercially on a large scale in service involving nitric acid,

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[1] G. C. Rogers, "Memo on Nitric Acid Corrosion in BSY," 21 Sept. 1965.

[2] D.A.G. Neet, "Radiation Exposure in the Switchyard," SLAC-TN-65-9, Jan. 1965.

and continue to be used in such installations. These first applications were of 15% to 18% Cr steel (now type 430) and soon thereafter of 182 Cr - 8% Ni steel (now type 304). The necessity for proper heat treatment to prevent accelerated corrosion and intergranular attack of these steels in nitric acid was demonstrated at once through service failures of improperly heat treated and as-welded equipment. These difficulties were eliminated by post-fabrication heat treatments involving slow cooling from about 1450°F for type 430, and rapid cooling from about 2000°F for type 304 stainless steels. Subsequently, for the austenitic grades, the use of stabilizing elements (particularly columbium in type 347) and, more recently, reduction of carbon content to 0.03% max (type 304L) have been effective in controlling this problem without the necessity for quenching fabricated equipment from a high-temperature heat treatment. In the as-welded condition, 304L and 347 show satisfactory resistance to corrosion by nitric acid and are therefore suitable for field-erected equipment.

Type 304 in the annealed and water-quenched condition has essentially the same resistance to corrosion by nitric acid as types 304L and 347, but type 304 should be heat treated after fabrication to prevent intergranular corrosion.

The stainless steels are relatively insensitive to factors such as the aeration, velocity and agitation of an aqueous fluid, since nitric acid is oxidizing and tends to favor passivity. Neither pitting nor stress-corrosion cracking is a problem under these circumstances. However, nitric acid causes integranular attack in in unstabilized stainless steels that contain more than 0.03% C, unless they have been properly heat treated. The presence of hydrofluoric acid in nitric acid, as in certain pickling solutions, increases such attack. Hydrofluoric acid also increases the rate of general corrosion, as do appreciable amounts of other halides.

In hot dilute mixtures of nitric and sulfuric acids, no appreciable attack occurs on the stainless steels when the ratio of nitric acid to sulfuric acid is about 2 to 1, or higher. This is one of a number of examples where sufficient nitric acid will prevent attack that would otherwise occur. With very dilute hot mixtures of sulfuric acid and nitric acid (about 1 to 1.5% total acid), where the proportion of nitric acid will not maintain passivity for the austenitic grades, type 443 (20% Cr, 1% Cu) has greater corrosion resistance.

Therefore, from a general corrosion standpoint, nitric acid proves to be more helpful than harmful when most stainless steels are considered. However, considering the real effects in the BSY some time after operation start, the accumulation of dirt (from dust and corrosion products) and moisture and the degradation of the alloy character by the beam may change the corrosion resistance of the alloy. Pitting corrosion, caused by the formation of areas where dirt causes the chemical stability of the oxide film on the stainless steel to be destroyed, may be encountered. Also, the alternate wetting and drying of components when the atmospheric humidity changes will serve to form localized concentrations of various acids. Accelerated corrosion can result from large differences in such electrolyte concentrations. Another corrosion mechanism that may be encountered includes pitting corrosion from alloy inhomogeneities in metal due to inclusions, coring, and distorted zones.

A by-product problem of using stainless steels is the cathodic (protected) nature of most stainless steels toward other anodic (corroded) materials. (Indium, used as the vacuum seal in the assembly, may be quite anodic to stainless steel.)

In addition to those corrosion problems mentioned above, dissolved oxygen in an acid solution can act as a cathodic depolarizer as it continuously removes hydrogen from a corroded zone. The removed hydrogen, which would otherwise retard galvanic corrosion by neutralizing the anodic/cathodic potential, will be replaced by newly-generated hydrogen by a continuous corrosion mechanism.

Stress-corrosion problems were thought to be sufficiently important to be considered separately.

Although the corrosion mechanisms mentioned above are but a few of the possible mechanisms which may be encountered as singular, or most probably as multiple, problems, the exact mechanisms which will be encountered in the BSY are impossible to predict. The very fact that most corrosion problems, specifically those related to material failures, are diagnosed after a material fails, illustrates the difficulty in predicting the exact problem and the relative ease of diagnosing failures. These seemingly-obscure corrosion mechanisms are included not to create any undue anxiety but to illustrate the many forms of corrosion and the difficulty in pinpointing the problem in a "simple" system.

2. Stress-Corrosion Cracking

Stress-corrosion cracking has been defined as the complex interplay of tensile stress and corrosion which leads to cracking in a metal or alloy. In the absence of a corrosive environment, the material exhibits normal load-carrying properties.

Tensile stresses must be present at the surface of the metal for stress-corrosion cracking to occur. Such stresses can be classified as either applied or residual. External loading inducing an applied stress in a member is easily visualized. Residual stresses, on the other hand, are developed in the metal by some operation in the metal's prior history which has caused heterogeneous yielding on the part of the metal. These residual stresses are divided into macrostresses and microstresses.

Macrostresses result when various components of an assembly restrain each other and may result also from nonuniform deformation incurred in fabrication operations. They may be developed also through heterogeneous deformation induced by thermal gradients in heat-treating operations.

Microstresses involve microscopic regions of a metal, such as grains and parts of grains. They also arise during forming operations, because individual grains do not deform with perfect homogeneity. The process of plastic deformation involves internal disturbances, such as slip, twinning, warping of crystal planes, orientation effects, and fragmentation of grains. Microstresses of atomic dimensions are associated with dislocations.

Of particular interest, from a metallurgical standpoint, are those reactions involving volume changes. In the case of the precipitation hardening steels, where a second phase is precipitated from a solid solution during cooling, each particle of precipitate will be compressed by the matrix if the second phase occupies a greater volume than the components from which it is formed. The matrix thus will be subjected to tensile stresses. Complicating factors are: 1) difference in coefficient of thermal expansion of different phases; 2) plastic flow in a hot and ductile interior; 3) suppression of phase transformation by a cooler rigid shell.

The quenching of steel from the austenitic state to form martensite also involves volume expansion. This results in the formation of the tetragonal lattice of martensite and the generation of an internal stress.

Other variables which often interact in stress-corrosion cracking are:

1) Alloy composition
2) Corrosive environment
3) Temperature
4) Time

Stress-corrosion cracking is not limited to an aqueous environment if attack by liquid metals, molten salts, and organics is considered in the broad classification of corrosion. Thus, cracking failures of brass in mercury, steel in molten zinc, stainless steel and titanium in molten chlorides, or titanium rivets in molten cadmium (from cadmium coatings) can be considered examples of stress-corrosion cracking.

Cracking in aqueous solutions is the most common form of stress-corrosion encountered. In addition to aqueous media, certain moist gases can promote this type of attack. However, most alloys are known for their cracking susceptibility in certain environments.

It should be noted that stress-corrosion cracking does not always occur in the solutions most commonly listed for each alloy. In fact, as mentioned previously, it occurs only under certain critical combinations of factors (alloy composition, stress level, temperature, time, and solution composition and concentration). The concentration of the solution has been shown to have a marked influence on the stress-corrosion cracking susceptibility of some alloys. Table I lists some of the environments in which martensitic stainless steels have failed by stress-corrosion cracking.

TABLE I

Environments in which Stress-Corrosion Cracking of Martensitic (Chromium) Steels has been Observed, Other Conditions Being Favorable
Nitrates
NaCl
Chlorides
Fluorides
Bromides
Iodides
Seacoast Atmosphere
Industrial Atmosphere
Water and Steam
H_2S

Dissolved oxygen in the solution can have an effect on stress-corrosion cracking. A well-known example is the increased susceptibility to stress-corrosion cracking of austenitic stainless steel in chloride solutions containing dissolved oxygen.

Cracking which occurs as a result of solution concentration can frequently be eliminated by design changes. Crevices which are possible sites for solution concentration should be eliminated wherever possible.

It is extremely difficult to predict when stress-corrosion cracking may develop in terms of projected service life of equipment. In fact, cracking failures in replicate specimens exposed under carefully controlled laboratory conditions often follow a statistical distribution curve, i.e., a few fail after a relatively short exposure and a few survive long-time exposure, while most specimens fail at some intermediate exposure time.

17-4 PH sheet material shows good resistance to stress-corrosion cracking in a marine atmosphere. Welding of the alloy in the highest strength condition (H 900) reduces the resistance to cracking. The standard solution heat treatment, following welding, apparently does not completely restore the resistance to stress-corrosion cracking of material. Therefore, in stress-corrosion environments it would appear to be safer to use material aged at higher temperatures, if the lower strength achieved under these conditions is acceptable.

In some instances, surface tensile stresses can be reduced by techniques such as shotpeening and tumbling which introduce compressed stresses in the surface. Corrosion, however, may remove this layer preferentially.

3. Hydrogen Embrittlement

Hydrogen embrittlement can become an important concern in the martensitic grades of stainless steel, generally increasing with hardness and carbon content. It becomes variable and less acute in ferritic steels, and is virtually unknown in the austenitic grades.

The embrittling hydrogen may be acquired as a result of the melting process, a heat treating atmosphere, or chemical and electrochemical processes such as pickling and electroplating.

Most heat-treating atmospheres contain hydrogen in the form of a) moisture, b) hydrocarbons, or c) elemental H_2 as an atmosphere or a dissociation product. The use of hydrogen or cracked ammonia for bright annealing in one plant was associated with cracking of wire coils of types 431 and 440C, although other plants have reported no similar difficulty. Nevertheless, it is possible that some loss in ductility may result from the bright annealing of <u>any</u> of the martensitic stainless steels.

Steel which is subjected to a tensile stress exceeding some critical value and which contains hydrogen that is free to move is susceptible to failure in a delayed, brittle manner. The problem is especially serious because the minimum stress for failure decreases as the strength of the steel is increased, and because failures occur with no appreciable ductility, even though in a tensile test the material may exhibit normal ductility. Under most conditions the strength level of the steel is the most important factor affecting the occurrence of delayed, brittle failure. Both the minimum applied stress that will result in failure and the time required for the failure to occur decrease as the tensile strength of the steel is increased. These failures occur in all types of steel microstructures except austenite. Alloy composition is a relatively unimportant factor in the hydrogen-induced, delayed brittle failure of body-centered cubic steels.

It has been shown that such failures depend directly on the hydrogen content of the steel, and the way in which the hydrogen gets into the steel is of no importance. Failures do not occur if hydrogen is kept out of the steel or is removed before the steel is damaged permanently. Normally, the critical amount of hydrogen required to induce failure is not present at the sites where failure initiates; hydrogen must move to these sites, either as the result of a hydrogen-concentration gradient or a stress gradient. The former condition prevails when the steel is exposed to an environment which permits hydrogen to enter its surface. Stress gradients that will cause hydrogen to move to regions of high tensile stress may result from bending or notches.

The problems in hydrogen analysis are very great as a result of the small amount usually present and the great mobility of hydrogen even at room temperature. Many investigations have relied upon such criteria as variations in cathodic charging time, variations in current density, variations in the concentration of, or time of exposure to, nonelectrolytic liquid environments (usually acids) as the basis for evaluating the effects of variations in hydrogen content. However, this is not easy to do because of the numerous processing operations that are potential sources of hydrogen and because of the very small amount of hydrogen (as little as 1 ppm, or possibly less) that can induce failure.

Table II lists some of the possible sources of hydrogen which may cause embrittlement in steels.

TABLE II

Sources of Hydrogen in Steel-Processing Environments

Steel-making operation – while steel is still molten

Fully killed steels where oxygen is removed which would normally combine with hydrogen

Liquid from rammed crucible refractories

Pickling operations

Cathodically-cleaned steel

Electrolytic machining

Electroplating

Moisture from water in coated welding electrodes

Heat treating at high temperatures in presence of low-pressure H_2

Cyclotron-proton irradiation

A representative 17-4 PH casting section was submitted for hydrogen analysis by the vacuum fusion technique. Results are not yet available.

C. Ring-Spring Material Selection

During the consideration of potential candidate ring-spring materials, several properties were considered. These were:

1) Room Temperature Yield Strength. As high a yield strength as possible, compatible with other considerations, is desired.

2) Room Temperature Toughness. A moderate degree of toughness is desired to withstand shock loads during assembly, etc.

3) Cost and Availability/Fabricability. A ring is required at reasonable cost commensurate with good properties. Casting is most desirable since other production techniques are either more expensive (closed-die forging, ring rolling) or compromise physical properties (welding). Machinability was also considered.

4) Corrosion Resistance. The resistance of candidate materials to the BSY environment and related problems were considered.

5) Weldability. Considered in the event that repairs or slight design modifications were required.

6) Radiation Damage Susceptibility. A difficult attribute to assign to material choices since limited quantitative information is available.

7) Other Effects. Susceptibility of choices to other normal processing environments, such as cleaning and degreasing solvents, electroplating problems (if required), etc.

A summary of the same considerations for the various alloy systems follows:

1) Titanium Alloys. Although these alloys exhibit good general corrosion and stress-corrosion resistance to the BSY environment and good room temperature strength (up to 175,000 psi yield strength), their high cost and fabricability problems (hard to properly weld) render them unsuitable.

2) Cobalt Alloys. These alloys are all too weak (80,000 psi yield strength) at room temperature. Besides high cost, the ultimate generation of copious amounts of Co^{60} also makes these alloys undesirable. Fabricability is also a problem.

3) Ferritic Stainless Steels. These steels are available and fabricable, but are too weak at room temperature to be suitable.

4) Austenitic Stainless Steels. The only technique to strengthen this class of steels above their moderate room temperature strengths is by cold working. 304 SS can be cold rolled to yield strengths up to 200,000 psi, but subsequent fabrication of plate (flame cutting, sawing, machining) raises costs appreciably. A possible exception is to ring roll by piercing and hot expanding a blank to size and then explosively forming to induce cold work. This relatively new technique is quite inexpensive but development time is not available. Austenitic castings are too weak for consideration.

5) Semi-Austenitic Precipitation Hardening Stainless Steels. PH 15-7 Mo and 17-7 PH steels are characteristic of this group and can be heat treated to yield strengths greater than 200,000 psi. However, the high susceptibility of these alloys to stress-corrosion cracking renders them undesirable.

6) Nickel-Base Alloys. Although somewhat more expensive than iron-base alloys, nickel-base alloys exhibit excellent strength, fabricability, and corrosion resistance in most environments. With yield strengths up to 170,000 psi, a nickel-base alloy would be an excellent choice. Lack of developmental lead time and relatively high cost render them a second choice to martensitic stainless steels.

7) Martensitic Stainless Steels. These steels are available as either quench-hardened or precipitation-hardened alloys. The quench-hardened alloys, like grades 420 or 440 can be hardened to yield strengths up to 195,000 psi (with some sacrifice in ductility or toughness), but their corrosion resistance and fabricability are impaired. The precipitation hardening alloys, of which 17-4 PH is a typical example, can be strengthened to 180,000 psi yield strength with a fair degree of toughness. Available in castings whose properties approach that of wrought material, these alloys are quite fabricable and exhibit good corrosion resistance. Although martensitic grades are susceptible to cracking by hydrogen embrittlement, only the austenitic stainless steels are not susceptible and these grades are not readily hardenable. Table III summarizes the effect of fast neutron irradiation on 17-4 PH. Changes in all mechanical properties can be noted, but are not extreme.

TABLE III

Effect of Fast Neutron Irradiation upon the Mechanical Properties of 17-4 PH Steel[3]

Property	Exposure	Unirradiated	Irradiated	Change	% Change
Yield strength, psi	2×10^{19}	144.7	179.4	+34.7	+23.9
Tensile strength, psi	"	148.5	181.0	+32.5	+21.9
% Elongation	"	16.0	13.0	-3.0	-18.7
% Reduction of Area	"	65.0	53.0	-12.0	-18.5
Hardness, R_c	"	26.0	32.0	+6.0	---
Yield strength, psi	1.3×10^{20}	144.7	194.5	+49.8	+34.5
Tensile strength, psi	"	148.5	197.0	+48.5	+32.7
% Elongation	"	16.0	12.0	-4.0	-25.0
% Reduction of area	"	65.0	44.0	-21.0	-32.3
Hardness, R_c	"	26.0	33.0	+7.0	---

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[3] DMIC Report 166, "The Effect of Nuclear Radiation on Structural Materials," Sept. 15, 1961, p. 38.

8) Other, Nonstandard Grades of Wrought Steels. Other grades of stainless steels, such as AM-350 and AM-355 are available with yield strengths and toughnesses similar to 17-4 PH. These alloys are also castable and weldable. Rene '41, Udimet 700, D-979 and Unitemp 212 are a series of Fe-Ni-Cr-Mo steels which all exhibit yield strengths greater than 125,000 psi. These alloys were developed for their high elevated-temperature strengths and in general are more expensive and difficult to machine than is 17-4 PH.

From this perusal of potential ring-spring alloys, it is concluded that 17-4 PH has the best combination of yield strength, toughness, availability, cost and fabricability. This conclusion was partially influenced by the fact that 17-4 PH was the ring-spring reference material and other materials were not sufficiently better to warrant a change. If other springs are made in the future, however, a more thorough investigation of other alloys, such as the nickel-base types, should be made. There was not enough time to completely investigate some alloy aspects, such as weldability, strength of casting versus wrought products, stress-corrosion susceptibility, radiation effects, etc.

D. Casting Vendor Talks and Processing Recommendations

The vendor who is producing the 17-4 PH ring-spring castings was visited and the results are reported.[4] Some of the recommendations from these talks include:

1) An improved inspection plan which details the radiographic and dye-penetrant inspection procedure.

2) The vendor was cautioned to not use halogen-containing solvents for cleaning and/or degreasing castings. Alcohol or acetone is a suitable substitute.

3) In view of the potential stress-corrosion problem with 17-4 PH the heat treatment was changed from H 900 to H 1025. Although this heat treatment reduces the yield strength from ≈ 180,000 to ≈ 160,000 psi, the material is more resistant to stress-corrosion

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[4] See Memo of G. P. Fritzke to R. Miksch, "Trip Report to Perfecto Cast Co., San Diego, Oct. 26, 1965," November 1, 1965.

cracking. The castings can also be machined after final aging with this heat treatment. Previously the castings were to be machined after solution heat treating but before aging at 900°F, and a shrinkage of ≈ 0.1% would be encountered. With the recommended sequence, there will be no dimensional changes after final machining.

4) Careful handling of the finished casting should be observed to prevent the formation of dents which act as stress risers. Identification of the casting by stamping of serial numbers in the surface is, of course, undesirable.

5) Avoid descaling in the 10% HNO_3 and 2% HF solution suggested in the brochure. Descaling in this manner would allow pickup of undesirable HF.

E. Casting Examination and Heat Treating Study

A representative casting in the solution heat-treated condition was metallographically examined to determine whether shrinkage porosity or dendritic segregation were problems. Several sections from critical portions of the casting were examined and it was noted (in eight samples) that shrinkage porosity was less than 1% in all instances. Cored dendrites (a phenomenon where alloy-rich metal is surrounded by alloy-poor metal - this situation can degrade corrosion resistance and the response of the alloy to heat treatment) were noted in all samples, however, and a small heat treating experiment was performed in an attempt to dissolve, or homogenize, these alloy-rich areas with the matrix alloy.

For the homogenizing study, several casting sections were obtained adjacent to each other to minimize sample differences. Some sections were then re-solution heat treated at 1925°F (to provide a double solution anneal) and other sections were heated to the homogenizing temperature of 2100°F. These latter sections were also solution heat treated and aged at 1050°F to provide comparison samples with the double-solutionized samples. The originally observed dendritic segregation was not appreciably affected by any of these heat treatments and therefore it would not be practical to attempt to homogenize the castings.

It is desirable to perform tensile tests on bars poured with each heat, however, to determine the change, if any, in expected mechanical properties due to this dendritic segregation.

III. SUMMARY AND CONCLUSIONS

From this study, several general conclusions can be drawn.

1) 17-4 PH stainless steel, heat treated to H 1025 is a suitable material for the ring-spring portion of the quick-disconnect vacuum coupling.

2) Corrosive environments, as they are now visualized, should not affect the ring-spring adversely. However, because of the great effect that minor variations in material quality, corrosion environment, etc., can have on the material, ring-spring failures will probably occur during accelerator lifetime. The cause of these failures will probably be due to some of the corrosion mechanism(s) discussed above.

3) No halogen-containing solvents should be used on the castings at any stage of processing.

4) The ring springs can be repair welded, but each instance should be handled as a special case to insure proper post-weld heat treatment, radiographic notation of weld area, careful visual inspection of the area, etc.

5) Materials with properties comparable to 17-4 PH can be used for the ring-spring material. However, no other material exhibits outstandingly greater reliability under BSY conditions.

6) A review of the casting vendor's facilities revealed good practices and a thorough understanding of our problem.

IV. RECOMMENDATIONS

In view of the critical role that the ring spring plays in the BSY design, several recommendations should be considered:

1) The present design calls for both male and female spring sections to be equally stressed. Since the male ring is "captured" by a flange and is difficult to replace, it is suggested that in a future design the male ring be overdesigned so the major stress load be carried by the female ring which is more easily replaced.

2) Several dummy assemblies should be positioned in the BSY where they can be subjected to conditions experienced by those in service. The dummies could then be examined periodically for possible failures and the results applied to those in service. As a part of this setup, it would be good to provide similar dummies in an area not subjected to the electron-beam effects so that samples could be periodically taken which would be divorced from beam effects. The assemblies in the BSY may become sufficiently radioactive to require hot-cell examination, whereas the "cold" dummies could be examined by conventional techniques.