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AN EVALUATION OF FIBERGLAS FOR USE IN
CUSHIONING PLUTONIUM PARTS

Ernest F. Lombardi

Robert I. Donovan

DOW CHEMICAL U.S.A.
ROCKY FLATS DIVISION
P. O. BOX 888
GOLDEN, COLORADO 80401

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DOW CHEMICAL U.S.A.
ROCKY FLATS DIVISION
P. O. BOX 888
GOLDEN, COLORADO 80401

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AN EVALUATION OF FIBERGLAS FOR USE IN CUSHIONING PLUTONIUM PARTS

Ernest F. Lombardi and Robert I. Donovan

Abstract. Fiberglas® cloth pads are used as protective cushions for plutonium parts in the fabrication lines. This material, because of its superior thermal and radiation stability, replaced chamois which had been used for this purpose for a number of years. Prior to implementing this materials change, a variety of experiments were performed with Fiberglas to evaluate the material for plutonium use. The results of this investigative program are presented and discussed in this report.

INTRODUCTION

This report summarizes the results of an investigative program which led to the use of Fiberglas® in the plutonium fabrication lines at Rocky Flats. The material is used as a protective cloth in stainless steel carriers for plutonium parts.

In past years, chamois was the most widely used material for this purpose, although air-blown polyethylene also was tried at one time. Both chamois and polyethylene have some undesirable properties for this type of application. For example, both materials are flammable and they are susceptible to radiolytic decomposition if in contact with a plutonium surface for extended periods of time. Because of these limitations, a nonflammable material with good radiation resistance was desired. Fiberglas was proposed because it appeared to meet both requirements, and in this respect, offered some particularly attractive advantages compared to most commercially available organic polymers or plastics.

FIBERGLAS: DESCRIPTION AND SOURCE

Fiberglas is the name given to many products such as fabrics and textiles which are composed of glass in fiber form. The glass structure consists of a lattice of inorganic oxides created at high temperatures. The material cannot burn, and it resists deterioration in highly corrosive environments.

The Fiberglas was supplied by the Owens-Corning Fiberglas Corporation Textile Development Laboratories

in Ashton, Rhode Island. Various samples of cloth were obtained for evaluation, but for our purposes, either of two different fabric styles appeared to be the most suitable. One was a smooth, plain weave cloth; the other a more decorative and rough looking fabric described as a cracked-ice weave.

Both fabric styles were composed of beta Fiberglas which is a descriptive term for glass fibers less than 4 microns in diameter. Reportedly, beta Fiberglas fibers are the finest and softest textile filaments known.

It was also requested that the Fiberglas be heat-cleaned by the supplier. This is a process by which any organic coatings or resins used in the manufacture of the fibers are burned off the finished product. Fibers that undergo this treatment are virtually pure glass. The residue content of heat-cleaned material is reported to be approximately 0.1 percent. The disadvantage of this finishing treatment is that it leaves the glass more susceptible to damage, but it was felt that organic residue in the cloth may present some plutonium contamination problems. The severity of any problem of this nature, of course, would depend on the quantity and type of organics used by the manufacturer and this information usually is proprietary and often difficult to ascertain experimentally.

The configuration of the glass pads ultimately adopted for production use consisted of six layers of heat-cleaned cracked-ice weave cloth, sewn into a circular pad approximately 17.5 inches in diameter.* This seemed to be the most desirable size and shape for the new lidded part carriers. Initial experience with the material in the lines uncovered some problems with fraying along the sewn edges of the cloth (fraying is an inherent property of Fiberglas). This problem was alleviated as much as possible by improving the stitching method. The outside edge of the cloth pad was bound with a heat-cleaned Fiberglas tape and then sewn with a heavier Teflon coated Fiberglas thread. Furthermore, all edges of the bias tape were folded under so that no raw edges were exposed.

*Manufactured by Unique Product Fabricators, Ashton, Rhode Island.

Additionally, and as previously mentioned, heat cleaning of the fabric does make the cloth more susceptible to damage, which in turn reduces the durability of the finished product. It may be possible to eliminate the heat-cleaning process and improve the durability of the fabric. This aspect of using the material will be further evaluated.

SUMMARY AND CONCLUSIONS

Analytical rinsing procedures showed that heat-cleaned Fiberglas contains very low levels of organic contamination. Under ordinary use conditions, the organic content of the cloth presents no foreseeable plutonium contamination problems. However, because of the softness of the glass fibers, care should be taken to minimize abrasive contact between plutonium and the Fiberglas. This action, depending on the degree of abrasion, could lead to some smearing of the Fiberglas onto plutonium. Should excessive abrasive contact be suspected, wire brushing or sanding the metal surface with Scotchbrite® should easily remove any contamination that may have resulted.

There is no measurable incompatibility between plutonium and Fiberglas under the conditions of anticipated use of the material. In laboratory tests, intimate contact of the materials for twenty-eight days produced no adverse effects to the plutonium. These experiments were performed in atmospheres of both vacuum and dry-box air. Additionally, operating experience with Fiberglas in the manufacturing lines further substantiates this conclusion. This has resulted in no known instances of part corrosion nor any other related problems which had compromised the integrity of the plutonium.

Extended exposure to radiation from a Cobalt-60 source produced no obvious adverse effects to the Fiberglas. The radiolytic gas production from the material was negligible and the glass fibers displayed no measurable differences in strength as a result of the gamma exposure. Most commercial polymers or thermoplastic materials (e.g., Teflon, polyethylene, polyvinyl chloride and others) would suffer almost complete physical deterioration under similar radiation exposure conditions.

Fiberglas is clearly superior to both chamois and polyethylene from the standpoint of temperature resistance. Comparative tests indicated that the Fiberglas was unaffected by temperature up to 700°C, whereas, both chamois and the polyethylene thermally degraded and burned at considerably lower temperatures. Additionally, samples of Fiberglas displayed no evidence of scorching

or ignition when laid directly on a small mass of burning plutonium hydride. The temperature of the burning mass was estimated to be about 810°C. The excellent heat resistance of the Fiberglas suggests a potential use of the material as a "blanket" for containing small plutonium fires, thereby preventing the spread to secondary combustibles.

Experience with a limited number of sample pads provided some important practical information about using Fiberglas in the manufacturing lines. This trial period resulted in an improved pad stitching method which reduced fraying of the glass fiber along seamed edges, and this in turn should increase the usable life of the cloth pads. Furthermore, it was found that rinsing the used contaminated pads with carbon tetrachloride reduces the contamination level of the material to less than plutonium discard limits. This was demonstrated by neutron and gamma counting of a sample population of pads both before and after the material had been rinsed.

EXPERIMENTAL DESIGN

The experimental approach was to evaluate the cleanliness of the Fiberglas cloth as received from the supplier, the compatibility of the cloth with plutonium under projected use conditions, and the effects of radiation on the material's properties; primarily, its strength and radiolytic gas production. Other tests were performed to verify some properties of the material such as its resistance to burning and its chemical durability. The final phase of the testing program involved a production scale evaluation with a limited number of Fiberglas pads. For all the various experiments, the Fiberglas was used in the as-received condition from the supplier.

ANALYTICAL EVALUATION OF FIBERGLAS

The cleanliness of the Fiberglas was determined by conventional analytical rinsing techniques using acetone, carbon tetrachloride, and trichloroethylene as solvents. Small samples of the cloth were treated individually with these solvents, and the soluble residue content was determined quantitatively. The residue was then analyzed by Infrared Spectrophotometry.

Table 1 summarizes the results of this analytical procedure. Both types of cloth contain a low level of organic contamination, although slightly more soluble

Table 1. Summary of Analytical Results.

Solvent Used	Mg Residue Per Gram of Fiberglas ^a (plain weave cloth)	Mg Residue Per Gram of Fiberglas ^a (cracked-ice weave cloth)
Acetone	1.0	0.56
Carbon Tetrachloride	0.62	0.56
Trichloroethylene	1.0	0.82

^aResidues comprised of a silicone oil plus hydrocarbon oil and organics containing a carbonyl functional group. The hydrocarbon oil was calculated for quantity as Texaco Regal A oil; the carbonyl compounds were calculated as dioctylphthalate.

residue was found in the plain weave material. These residue levels agree favorably with the supplier's claim about the cleanliness of heat-cleaned Fiberglas and, from the practical standpoint, the organic content of the cloth should present no foreseeable problems with respect to contaminating plutonium. The residues were tentatively identified as a silicone oil plus some aliphatic hydrocarbon oil and other organic compounds containing a carbonyl functional group.

To complement the solvent rinsing procedure, samples of fiberglas were also heated *in vacuo* to several hundred degrees centigrade, and the gas evolution monitored by TOF mass spectrometry. Under these conditions, only trace amounts of gas were produced from the cloth samples. But a noticeable difference in the temperature for the onset of outgassing was observed. The cracked-ice weave material began to outgas as low as 75°C, whereas the plain weave material was heated to over 200°C before any measurable gas production was observed. The outgassing products appeared to be the same for both Fiberglas weaves. The gaseous components were tentatively identified as mixtures of an organic ester, aliphatic hydrocarbons (up to 9 carbons) and compounds in the alkyl benzene class. There was no indication of any volatile silicone compounds.

PLUTONIUM COMPATIBILITY EXPERIMENTS

The plutonium compatibility tests were performed by toting small, delta-plutonium disks in direct contact with the Fiberglas for twenty-eight days and then analyzing the metal surfaces for evidence of any

abnormal corrosion. X-ray diffraction was used as the analytical tool. The tests were conducted at ambient temperature in atmospheres of vacuum and circulating glovebox air. Control experiments were conducted for the two different atmospheres. The results were the same for all disks analyzed. Regardless of whether or not the disks had been exposed to the Fiberglas, the major surface constituents were Δ Pu with 150-200 Å of PuO₂. These data indicate that under the conditions tested, there is no measurable incompatibility between the two materials.

An additional consideration with regard to plutonium use was the possibility of the Fiberglas abrading or marring a plutonium surface. There are actually two parts to this question. One is concerned with the cloth, because of its softness, depositing onto plutonium if the two materials are in firm contact or rubbed together. The other is the possibility of the Fiberglas actually scratching plutonium.

Two relatively simple tests were performed to evaluate the smearing potential of Fiberglas. First, a sample of the cloth was vigorously rubbed against a plutonium disk that had been wire brushed and analyzed for surface contaminants by Spark Source Mass Spectrometry. Following this treatment, the cloth-rubbed surface was reanalyzed. This same analytical approach was used for the second test, but in this case the Fiberglas cloth and a plutonium disk were pressed together and held in this configuration for 88 hours without any abrasive action.

Tables 2 and 3 summarize the analytical results for the two separate tests. This information indicates that abrasive action produces some smearing of the Fiberglas onto plutonium (Table 2). This can be seen particularly by the increases in the concentration of elements boron, calcium, magnesium, and silicon, all of which are major elemental constituents of the Fiberglas. However, firm contact between the two materials without any abrasive action resulted in very little change in the concentration of surface impurities on the plutonium.

The chances of the glass fabric scratching the plutonium can be virtually discounted. This was demonstrated by microscopic examination of a polished plutonium surface, both before and after the surface had been vigorously rubbed with the cloth. There was no evidence of any surface abrasion.

The results of these experiments indicate that abrasive contact between the Fiberglas and plutonium could

Table 2. Results of Plutonium-Fiberglass Abrasion Test.

Type of Fiberglass	Plutonium Surface Treatment	Surface Constituents, Atom Percent of Surface Sparked. ^a									Other Elements ^t
		Al	B	Ca	Fe	Mg	Si	O	C	Pu	
Plain Weave Cloth	Wire Brushing	0.2	—	0.09	7.3	0.9	0.6	2.1	8.6	63.7	16.6
	Vigorous Rubbing with Fiberglass Cloth	0.5	0.1	1.0	1.5	3.4	2.6	1.7	13.4	63.4	12.4
	Wire Brushing Again	0.2	—	0.2	0.2	1.4	0.3	0.9	0.8	91.0	5.1
Cracked-Ice Weave Cloth	Wire Brushing	0.2	—	0.07	0.5	0.4	0.3	1.5	5.5	85.0	6.6
	Vigorous Rubbing with Fiberglass Cloth	0.2	0.02	1.0	0.2	3.3	1.3	1.0	7.6	75.0	10.3
	Wire Brushing Again	0.1	—	0.3	0.4	1.8	0.6	1.0	3.2	87.9	4.7

^a A dash (—) indicates none detected.^b This column includes total of elements Be, Cl, Cr, Cu, F, Ga, K, Mn, N, Na, Ni, P, S, and Zn.

Table 3. Results of Plutonium-Fiberglass Weighted Contact Test.

Type of Fiberglass	Plutonium Surface Treatment	Surface Constituents, Atom Percent of Surface Sparked. ^a									Other Elements ^b
		Al	B	Ca	Fe	Mg	Si	O	C	Pu	
Plain Weave Cloth	Wire Brushing	0.4	—	0.3	3.2	1.0	0.6	2.8	7.5	71.9	12.27
	Weighted Contact with Fiberglass Cloth, No Rubbing Action	0.1	—	0.2	0.6	1.2	0.6	1.0	7.2	84.1	5.04
Cracked-Ice Weave Cloth	Wire Brushing	0.2	—	0.1	0.5	0.6	0.2	1.5	2.7	90.6	4.2
	Weighted Contact with Fiberglass Cloth, No Rubbing Action	0.1	—	0.09	0.3	0.6	2.3	0.9	2.9	87.0	5.8

^a A dash (—) indicates none detected.^b This column includes total of elements Be, Cl, Cr, Cu, F, Ga, K, Mn, N, Na, Ni, P, S, and Zn.

lead to some plutonium contamination. However, it is important to note that the results shown in Table 2 represent an extreme case from the production standpoint, since it is very unlikely that any plutonium part would ever be subjected to such vigorous abrasive treatment. But, despite this fact, care should be taken to minimize any chance of the Fiberglass abrading against the plutonium. Should abrasive contact be suspected, the analytical evidence indicates that wire brushing the plutonium adequately removes any con-

tamination that may result from the Fiberglass. Sanding the metal surface with Scotchbrite® also should be an effective alternative cleaning procedure.

RADIATION EXPOSURE TESTS

The radiation exposure tests utilized a Gammacell 220®, which is a commercial source containing about 3500 curies of Cobalt-60. Actually, under use condi-

tions, the Fiberglas would experience plutonium alpha radiation (i.e., the absorbed dose from plutonium gamma rays is negligible compared to that from alpha particles), but for the nature of these experiments, any effects caused by the Cobalt-60 gamma energy can be used to predict the alpha effects with a reasonably high degree of confidence. This is true because both the plutonium-239 surface and the Cobalt-60 gamma source have approximately the same dose rate. The difference is that the alpha particle energy is deposited to a depth of only about 40 microns, whereas the gamma energy is deposited throughout the Fiberglas material. Using this line of reasoning and a measured Fiberglas cloth thickness of approximately 200 microns, one can estimate that an hour in the Gammacell roughly approximates that amount of radiation that the cloth would experience during five hours in contact with a plutonium surface.

The first experiment from the radiation standpoint was to evaluate the radiolytic gas production from the Fiberglas. This was done by encapsulating weighed samples of the Fiberglas in evacuated Pyrex® glass cells, irradiating the loaded cells for 281 consecutive hours, and then analyzing the gaseous products by Mass Spectrometry. Table 4 summarizes the gas results from two separate tests, both of which used samples of the cracked-ice weave cloth. The principal radiolysis product was carbon dioxide. The next most prevalent products were hydrogen in one case and methane in the other. Additionally, both tests exhibited traces of ethanol, acetic acid, and organic alkanes up to four carbons.

Table 4. The Radiolytic Gas Production from Fiberglas.

Gaseous Products	Micromoles per gram Fiberglas	
	Sample-1	Sample-2
H ₂	0.139	0.109
N ₂	0.052	0.048
O ₂	0.006	0.006
A	0.0004	0.0004
CO ₂	0.288	0.237
CH ₄	0.175	0.079
C ₃ H ₈	0.133	0.012
C ₄ H ₁₀	0.005	0.005
C ₂ H ₅ OH	0.019	0.012
CH ₃ COOH	0.007	0.0004
Total Gas Product	0.8244	0.5088

The results of these experiments indicate that the radiolytic gas production from the Fiberglas is comparatively low in terms of the high total dose that the material received. From this point of view, gas evolution can be considered practically negligible, and it is extremely doubtful that radiolysis of the Fiberglas could lead to any significant plutonium corrosion in the manufacturing lines.

The effect of gamma radiation on the strength of the beta Fiberglas also was evaluated. The experimental approach was to separate multifilament strands from the cloth, irradiate some of the strands for 214 hours in air, and then compare the breaking strengths of the irradiated versus the unirradiated strands.

The breaking strength was determined by a tensile testing procedure developed especially for this purpose. The technique utilized approximately ten-inch specimens, the ends of which were epoxied to a strip of paper. The center four inches of the strand was unepoxied. The epoxy fulfilled two purposes; first, it bonded the filaments together to prevent the interior filaments from pulling out. Second, it bonded the strand to a backing material which made for easier handling and permitted the strand to remain straight after the epoxy cured. The test procedure was to insert the paper strip into the testing apparatus with epoxied ends of the glass fiber inside rubber-faced friction grips. The distance between the grips was approximately four inches. The strip of paper then was cut in two and the test performed at a cross-head speed of 0.05 in./minute, which corresponds to a strain rate of 0.0125 in./in./minute. The results were obtained from autographic records of load and time. Load measuring accuracy was about 0.1 pound.

The results of eight separate tensile tests are shown in Table 5. This information indicates that there is no

Table 5. Breaking Strength of Irradiated versus Unirradiated Fiberglas Filaments.

	Breaking Load (pounds)	
	Unirradiated Strands	Irradiated Strands
	3.1	3.0
	2.9	2.8
	2.6	2.6
	3.0	3.5
Average	2.9	3.0
Standard Deviation	0.22	0.39

significant difference in breaking strength between the irradiated and unirradiated specimens. It can be concluded that this level of irradiation does not appear to weaken the glass fibers.

FLAMMABILITY TESTS

The temperature resistance of Fiberglas was compared to both that of chamois and polyethylene foam, two materials which have been used for cushioning plutonium parts in the carriers. This test was done primarily to demonstrate the clear superiority in burning characteristics of the Fiberglas.

The experimental approach was to heat the materials of interest in a muffle furnace and record indications of physical changes such as melting, smoking, or degradation and ignition. The chamois began to smoke at a furnace temperature of 300°C and the material ignited at 525°C. The polyethylene first melted, then began to emit smoke and ignited at 560°C (furnace temperature) producing a very hot flame, the peak temperature of which was recorded as 850°C. On the other hand, the Fiberglas was unaffected by furnace temperatures of up to 700°C. The glass displayed no indication of melting, smoking or ignition during the course of the heating period.

To complement this information, strips of Fiberglas cloth also were laid directly on a small mass of burning plutonium hydride. The temperature of the burning mass was estimated to be 810°C. The Fiberglas material was unaffected by this treatment. There was no evidence of scorching or ignition of the cloth strips. The excellent thermal stability of the Fiberglas may suggest a potential use of the material as a "blanket" for preventing the spread of small plutonium fires in the manufacturing lines.

OTHER USE-RELATED EXPERIMENTS

Most materials in the fabrication lines are exposed at one time or another to the various solvents and oils used for plutonium processing. This would be particularly true for any part-cushioning material, since it would be in intimate plutonium contact.

To demonstrate the excellent chemical resistance of Fiberglas, samples of the cloth were immersed for periods of over two weeks in carbon tetrachloride, perchloroethylene, trichloroethylene, acetone, and 8N nitric acid. The organic solvents were kept at

room temperature, but the nitric acid was heated slightly for brief periods of time. At the conclusion of these soaking periods, the Fiberglas displayed no obvious adverse effects. The textile fibers did not appear to deteriorate, swell, or undergo any significant chemical change.

Tests were also conducted to evaluate the oil absorbency characteristics of the Fiberglas. This was done because experience with chamois indicated that the material over a period of time became fairly saturated with machining oil, which in turn, could lead to some plutonium corrosion problems. Weighed samples of both chamois and Fiberglas were immersed in Texaco Regal Oil A® for about one minute, then each was suspended overnight in air to allow oil drainage. Following this, the samples were reweighed to determine the amount of oil retention. The weight data indicate that the Fiberglas absorbed and retained considerably less oil than the chamois. For chamois, the weight increase because of oil pickup, was 4.91 grams per gram of original material. This can be contrasted to weight increases of only 1.35 and 1.56 grams per gram for the two respective fiberglas cloth weaves, plain and cracked-ice.

PRODUCTION SCALE EVALUATION

Before replacing chamois with Fiberglas, a production scale trial was conducted with fifty sample pads of each type weave (plain and cracked-ice weave). This was done primarily to demonstrate the feasibility of using Fiberglas in the lines and also to assess any operating difficulties with the material that may not have been obviated from laboratory scale testing. The main criteria of interest was evaluating whether the used pads would contain recoverable amounts of plutonium as well as the feasibility of cleaning and reusing the material.

The sample pads were introduced into the drybox system and each pad accompanied a plutonium part through the normal fabrication sequence. The cloth pads then were removed from the carriers, separated according to type weave, and stored in covered containers until all pads had been collected.

Twenty-five pads of each type weave were removed from the line and submitted for neutron and gamma counting to determine if they contained recoverable amounts of plutonium. The counting results indicated that both sample populations were plutonium contaminated greater than the discard level. The results,

in terms of total grams plutonium per gram of Fiberglas, were 7.5×10^{-4} for the plain weave cloths and 9.6×10^{-4} for the cracked-ice weave material. Since the cracked-ice material contained slightly more contamination, these twenty-five pads were put back into the fabrication line, rinsed with carbon tetrachloride, air dried, bagged out, and again counted for plutonium content. The results after rinsing indicated that the contamination level of the Fiberglas had been reduced about threefold. The resultant assay was 3.6×10^{-4} grams plutonium per gram of Fiberglas, which is considered to be less than discard limits. On the basis of this information, it appears that a simple rinse with carbon tetrachloride effectively removes residual contamination from the cloth pads. However, despite these favorable results, additional work is being directed toward developing a recovery process for very low levels of plutonium contamination.

Some of the sample pads then were reused in the fabrication lines. This action uncovered some problems. The most notable deficiency was an inadequate stitching method which led to fraying along the seamed edges of the Fiberglas. The stitching method was improved for the cloth pads currently being used in the manufacturing lines and this has increased the usable life of the pads.

The fact that the Fiberglas pads can be reused is an additional advantage over chamois. Each chamois was used only once, then replaced. Cleaning and reuse of the material was not practical, since it removed some of the natural oils and this resulted in a stiff hardened skin no longer suitable for plutonium use. The Fiberglas cloths are more expensive initially (\$3.77 each as opposed to \$1.80 per chamois), but, since these can be reused, costwise they would be cheaper than the chamois.