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Investigation Into Radiation-Induced Compaction of Zerodur™

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Marshall Space Flight Center • MSFC, Alabama

National Aeronautics and Space Administration
Marshall Space Flight Center • MSFC, Alabama 35812

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INTRODUCTION

The space environment has historically been detrimental to materials functioning in space. The life of a spacecraft is directly proportional to the ability of its materials to maintain functional status. A material's life in a specific environment is best determined by real-time exposure in that environment. Extended life missions reduce the practicality of real-time testing and rely on accelerated ground-based testing and modeling to generate material life predictions. This report focuses on ground-based testing of Zerodur™ and the subsequent predictions of compaction as a function of proton radiation fluence and energy.

Zerodur™ is a glass-ceramic material composed of 80-percent lithium aluminum silicate crystal (β eucrypite and β quartz) and 20-percent high silica glass. Since the coefficient of thermal expansion (CTE) of the mixed crystal is negative and that of the glass is positive, a zero thermal expansion coefficient can be achieved by properly adjusting the relative concentrations of the crystalline and glass phases.[1] A zero thermal expansion coefficient material is well suited for many space applications when extreme optical figure stability is required. Zerodur™ is baselined for use as an optical substrate in the Advanced X-ray Astrophysics Facility (AXAF-I). The Zerodur™ optical substrate must be dimensionally stable over the range of environmental conditions in which the AXAF-I operates. Zerodur™ and other low CTE materials have been shown to be dimensionally unstable when exposed to large doses of ionizing radiation.[2]

EXPERIMENTAL PROCEDURE

The Zerodur™ samples used in this investigation were 3-in diameter disks with a thickness of 0.25 in. Each sample surface was analyzed using a noncontact optical interferometer (WYKO)™ prior to irradiation. All samples were supplied by and polished by MSFC to a nominal 1-nm root-mean-square (RMS) surface flatness.

Zerodur™ is formulated by the manufacturer to be dimensionally stable over the temperature range from 0 °C to 100 °C.[3] Knowing that radiation exposure will elevate sample temperature, the first test on Zerodur™ was determining the radiation induced temperature change caused by proton irradiation as a function of time.

The sample temperature will vary directly with energy deposited into the sample. Energy deposition varies directly with beam intensity. A Zerodur™ sample was exposed to a 2.0-MeV proton beam current of 1.75×10^{11} protons/cm^{2}s. This beam current was over twice as intense as the beam current used when investigating the radiation-induced compaction. The Zerodur™ temperature was monitored by an infrared thermography system. Results from this test, shown in figure 1, indicate that after 1 hour of exposure the maximum temperature observed was 40 °C. Temperature effects are considered negligible for this investigation since the 40 °C maximum surface temperature observed is less than the 100 °C stability limit.[3]

The proton-induced compaction exposures were performed in the Combined Environmental Effects Test Cell 2 (CEETC2) system which is described in NASA TM 108416.[4] This system utilizes a raster scanner that rasters the proton beam into a divergent pattern and provides less than 3-percent variation of charge across the sample surface. A uniform deposition of charge eliminates localized intensity variations. A sample holder, shown in figure 2, was fabricated specifically for this series of tests.

The Zerodur™ sample was enclosed in the masking holder, which allowed radiation to interact with a small region of Zerodur™ surface. A Faraday cup was included in the sample holder to integrate, in-situ, the charge deposited on the sample. The proton flux was minimized to reduce any sample heating effects. The flux utilized for all proton exposures was 5×10^{10} protons/cm^{2}s (1.5×10^5 rads/s).

A test was performed to determine if compaction would be a function of flux levels planned for this experimental investigation. Results of this test demonstrated that the flux contribution to the radiation-induced compaction is significant only at high flux levels, as shown in figure 3. This test utilized 2.0-MeV protons and maintained a constant fluence of 1.74×10^{14} protons/cm^2. The rate at which the fluence was applied ranged from a flux of 1.7×10^{10} to a flux of 2×10^{13} protons/cm^{2}s. This test shows the compaction to be constant over the flux range from 5×10^{10} to 1×10^{12} protons/cm^{2}s.

At flux values above 1×10^{12} protons/cm^{2}s, the compaction increases a function of the dose rate. The results of this test, combined with the desire to eliminate sample heating, provided justification for using a low flux level to investigate compaction.

The proton-induced compaction exposure portion of this investigation utilized beam energies of 0.5, 2.0, and 2.5 MeV. Proton fluences ranged from 1.5×10^{11} to 1.4×10^{15} protons/cm.^{2} Table 1 summarizes the proton exposure parameters and the corresponding radiation induced compaction. Figure 4 graphically shows the relationship between proton dose and compaction.

Radiation-induced compaction was measured by two methods, noncontact optical interferometry (WYKO™) and a stylus-type surface profiler (DEKTAK II™). The Zerodur™ sample, shown in figure 2, was partially masked to prevent the entire sample from being exposed to the incident radiation. A physical interface was formed during irradiation between the exposed region of the sample and the unexposed or masked region. Proton radiation exposure formed a depression that increased in magnitude with increasing radiation dose. Compaction, as described in this report, was the magnitude of this depression.

RESULTS

The results of the proton exposure tests are shown in figure 4. All curves are power law functions of the form,

$$C = AD^B ,$$

where C is the radiation induced compaction in angstroms, D is the radiation dose in rads (Si), and A and B are constants. A power regression curve fit of the form,

$$\ln C = A+(B)(\ln D) ,$$

was utilized to determine the values of A and B, which are the intercept and slope values from the power regression. Table 2 shows the calculated values for A and B.

Higby et al.[2] investigated the radiation induced compaction of several low CTE materials, including Zerodur™. One result of Higby's work was that compaction has a power law dependence on radiation dose and has the form,

$$\Delta\rho/\rho = AD^{B} .$$

Density is given by ρ in g/cm^3. A and B are constants, and D is the dose in rads (Si).

Using a standard linear regression, Higby et al. determined the values of A and B to be,

$$A = -4.52 \pm 1.022 \quad \text{and} \quad B = 0.38 \pm 0.124 \ ,$$

for Zerodur™ exposed to 2.0-MeV electrons.

The radiation-induced compaction results of this current experimental investigation and those of Higby are similar even though the incident radiation is different. Efforts to directly relate the two power law relationships have not yielded satisfactory results. The authors believe the reason for the unsatisfactory results is due, in part, to the difference in the type of incident radiation. Another series of tests using electrons as incident particles to irradiate Zerodur™ is planned to resolve this apparent discrepancy. The goal of this electron exposure test is to determine if a unified relationship between compaction and radiation dose can be obtained.

CONCLUSIONS

This investigation indicates that Zerodur™ will experience a dimensional change (compaction) when exposed to proton radiation. Ruller and Friebele[5] discuss possible compaction mechanisms when silica materials are irradiated. These mechanisms reduce to the fact that irradiation creates displacements, electronic defects, and/or breaks the Si-O-Si bonds.

Radiation damage theory states that the incident particle does not produce many displacements, but rather initiates a collision cascade[6]. The energy of the primary knock-on atom (primary atom in the collision cascade) is obtained from the collision of the incident particle and the stationary atom. Energy transferred to the stationary atom is dependent on the mass and energy of the incident particle. Since protons are approximately 1,000 times more massive than electrons, the number of displacements that can be produced by protons is much greater than that produced by electrons of equal energy. The results of this investigation are insufficient to determine the primary cause of compaction. The series of tests using electrons as the incident particles is required to begin to formulate conclusions about the primary cause of compaction. According to Ruller, displacements, electronic defects, and breaking the Si-O-Si bonds all produce the same macroscopic effect. The effect described by Ruller for Zerodur™ is that the structure will relax and fill the relatively large interstices that exist in the interconnecting network of silicon and oxygen atoms, causing compaction.[5]

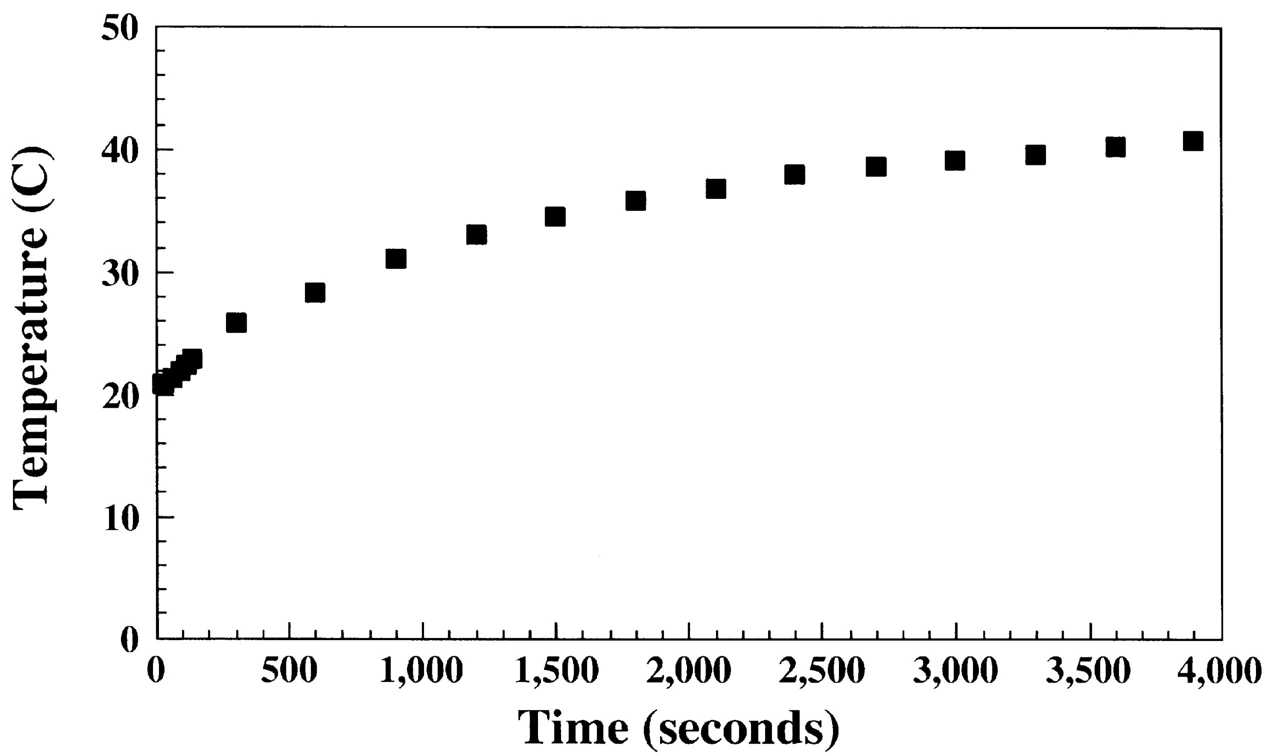

Figure 1. Increase in Zerodur™ surface temperature exposed to 2.0 MeV protons at 175×10^{11} protons/cm^{2}s.

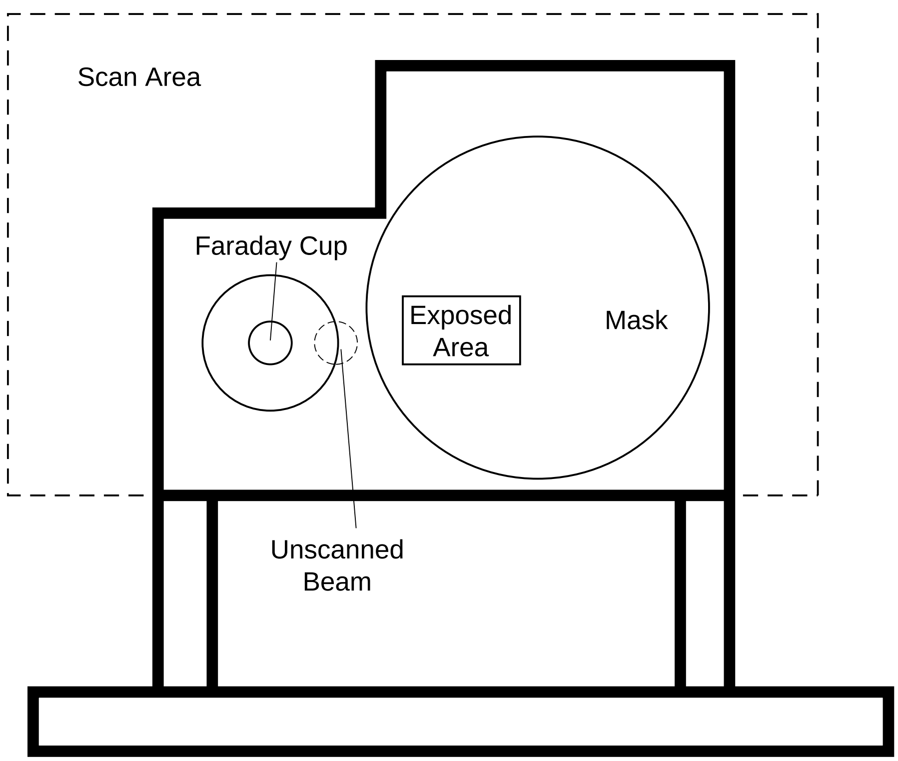

Figure 2. Zerodur™ sample holder in test configuration.

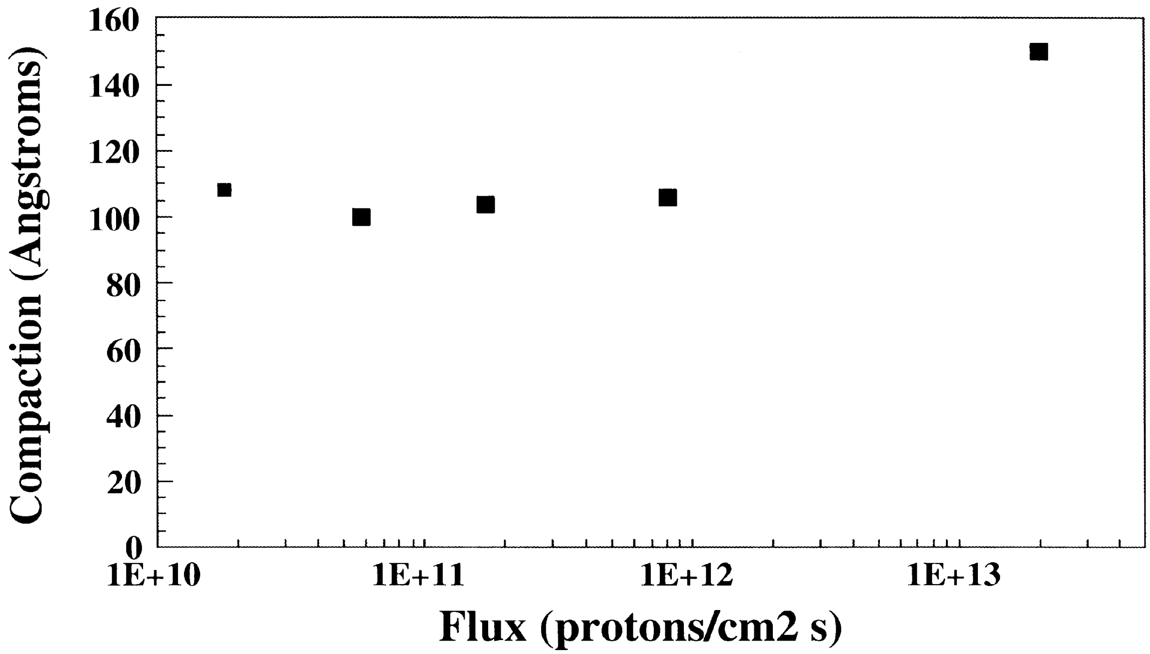

Figure 3. Relationship between the compaction of Zerodur™ and 2.0 MeV proton flux. The fluence for this test was 1.74×10^{14} protons/cm^2.

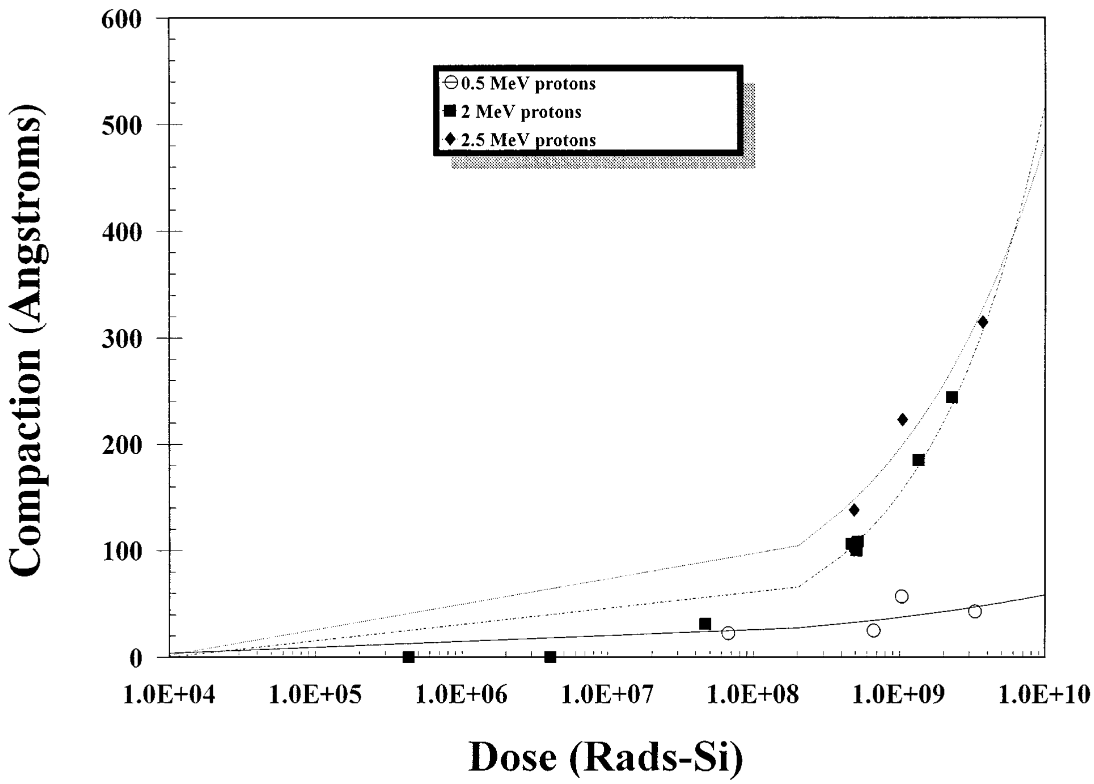

Figure 4. Proton-induced compaction of Zerodur™ exposed to a flux of 5×10^{10} protons/cm^{2}s.

Table 1. Summary of proton-induced compaction exposures.

Energy (MeV)	Dose (Rads)	Fluence (protons/cm^2)	Flux (protons/cm^{2}s)	Measured Compaction (Å)
0.5	6.7E+7	1E+13	4.12E+10	22.5
0.5	6.7E+8	1E+14	4.54E+10	25
0.5	1.04E+9	1.56E+14	5E+10	57
0.5	3.3E+9	4.9E+14	4.84E+10	42.8
2	437,000	1.5E+11	3.75E+9	0
2	4,080,000	1.4E+12	4.7E+9	0
2	4.7E+7	1.6E+13	5.4E+10	31
2	5.1E+8	1.74E+14	5.8E+10	100
2	5E+8	1.7E+14	1.7E+11	104
2	1.36E+9	4.66E+14	5.82E+10	185
2	4.75E+8	1.63E+14	8.15E+11	106
2	5.2E+8	1.77E+14	1.78E+10	108
2	5.82E+8	2E+14	2E+13	150
2	2.3E+9	7.89E+14	4E+10	244
2.5	4.9E+8	1.85E+14	4E+10	138
2.5	1.05E+9	3.95E+14	5E+10	223
2.5	3.715E+9	1.4E+15	9.867E+10	315

Table 2. Calculated values of the constants *A* and *B* determined from a power regression curve fit.

Proton Energy	*A*	*B*
0.5 MeV	–0.39	0.194
2.0 MeV	–5.96	0.53
2.5 MeV	–2.8	0.39

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