REPLICA GRATING STUDY

NGR-22-091-002

INTERIM REPORT

June 30, 1968 - December 31, 1968

College of the Holy Cross
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Abstract

Preliminary thermal vacuum in-situ testing has started using a new refrigerant with thermal range in excess of -90°C to +150°C.

The trial tests, using an obsolete replica grating of known limited thermal range, showed a deterioration in beamwidth at both 1216A and 4471A increasing slowly until 55°C and thereafter deteriorating more rapidly. A cooling back to the initial 25°C did not show an "elasticity" in the sense that the beamwidth did not return to the initial low value. Testing at reduced temperatures will be undertaken as soon as a new pump capable of handling the relatively high viscosity of the refrigerant at the lower temperatures is delivered.

Stress by energetic radiation has continued and the range of materials has been expanded to include virtually all those now used for making replica diffraction gratings. These materials are Pyrex, BSC2, synthetic fused silica, fused quartz, Vycor, and Cervit. Cervit, while possessing a very low thermal coefficient of expansion, curiously enough shows an exceptional sensitivity to electron irradiation. Synthetic fused silica at this point is indicated as showing the least deformation to electron irradiation. Beta irradiation of Pyrex by selected radioisotopes has continued.

1. Purpose of Effort

1.1 Thermal Vacuum Stressing

After many equipment failures, the thermal vacuum in-situ stressing was started. The purpose of this work, as has previously been stated, is to observe the response of the gratings while they are actually being stressed. Previous thermal vacuum tests have been made but these demonstrated only the ability (a kind of "elasticity") of the gratings to return to their unstressed behaviour rather than what they actually did while being stressed.

1.2 Particle Irradiation

The purpose of the irradiation work this period was to extend our knowledge of the response to a space environment involving high energy electrons of replica gratings and substrates of important but hitherto untested materials. Accordingly Cervit (a vitrified ceramic), GE151 (a high grade synthetic fused silica), GE125 (a fused quartz), and Vycor were subjected to high energy electrons from a Dynamitron accelerator. The low intensity irradiation by special radioisotope sources (see description in Phase IV Report) of Pyrex gratings and substrates was continued.

2. Nature of Effort

2.1 Thermal Vacuum Stressing

In the last report a difficulty with the R-11 refrigerant was noted, viz, that it boiled at room temperature unless pressurized. A different refrigerant, TMC, was tried and it indeed did extend the allowable thermal range to include room temperature and unpressurized operation. Unfortunately after using the TMC for several weeks it was found that in spite of pump manufacturer's guarantees to the contrary, the TMC was attacking the pump seals and producing leaks.

Investigation of a better brine with which to thermally stress gratings in a vacuum has now led us to the use of a relatively new refrigerant, Dupont's E-3. We gratfully acknowledge the gift on an initial charge of the E-3 from the Dupont Co. This brine, while quite expensive, will cover the desired thermal range and do it without pressurization. One problem remains at the lower temperatures and that is the rapid rise in viscosity of the brine. At -68°C the viscosity is 75 centistokes, while at 100°C it is only about 0.4 centistokes. The present centrifugal pump is not capable of sufficient pressure at the lower temperatures to push the brine fast enough to lower the temperatures in the test bed to a satisfactory point. Thus while a new pump is being procured, attention has been concentrated on testing at elevated temperatures.

In view of the fact that we planned to test the concave gratings to the point where the surfaces were deformed beyond use, it was felt desirable to test our procedure on an old grating made by a replication process long since discarded.

Two wavelengths were selected for test in order to get a feel as to whether the tests of gratings designed for use in the vacuum ultraviolet could be adequately measured in the visible region. The results are shown in Table 1. The readings were taken as the temperature was increased from 25°C to the point where a gross deterioration in the 1216A line occurred then the temperature was allowed to cool back to 25°C and the grating checked again. Grating temperatures were measured by means of a thermocouple affixed to the side of the gratings with Devcon--a previously tested procedure.

TABLE I

Variation of Beamwidth and Maximum Instensity as a Function of Replica Grating Temperature

A. 1216A

Temperature (°C)	Beamwidth (BW) (Half-power Points)	Maximum Intensity (cms Deflection)
25	.29A	71
35	.32	66
45	.32	58
55	.36	54
65	gross	32
25 - line could not be found - submerged in noise		

B. 4471A

Temperature (°C)	Beamwidth (BW) (Half-power Points)	Maximum Intensity (cms Deflection)
25	.32A	72
35	.35	65
45	.33	78
55	.33	54
65	.39	34
25	.43	22

Certain observations can be made with respect to Table I.

1. The BW are accurate to +.02A as deduced from previous experiments.
2. As the temperature is increased, the BW broadens slowly until the temperature hits 55°C. After this point the deterioration rapidly increases.
3. This old replication material is not "elastic", i.e. it does not return to its original condition after the thermal stress is removed. If anything, there seems to have been a "deformation inertia" in that the BW when measured after the temperature was reduced is <u>worse</u> than it was at the elevated temperature from which it was cooled.
4. The "maximum intensity", i.e. the maximum deflection of the photomultiplier output recording pen as the test bed swept through the wavelength under study, definitely reduced as the temperature went up.
5. Efficiency measurements were made at each temperature but we are not completely satisfied with the technique used and more work remains to be done before full credence can be given the results.

2.2 Particle Irradiation

2.2.1 Irradiation by Radioisotope

In spite of the fact that the "total" dose, i.e. integrated dose over the complete energy spectrum of the isotopes used, was about 10^{15} electrons, little or no deformation of the surfaces of either gratings or aluminized substrates was noted. The substrate material was Pyrex, a material which if given a monoenergetic dose of this same amount in a relatively short time would have deformed perhaps as much as 2 fringes.

Two possibilities immediately present themselves (a) the surface deformation is energy sensitive and/or (b) the surface deformation is rate sensitive. There is some evidence to support the deformation being energy sensitive as Dynamitron irradiations of constant dose at 10^{14} electrons/cm^2 produce more distortion at 1.0 Mev than at .3 Mev on the basis of preliminary tests. While energy dependence as well as rate dependence does appear likely, more work will have to be done to settle this point.

It should be definitely emphasized that this question of the deformation being energy dependent is indeed an important point as in space we have a poly-- rather than a mono-energetic environment.

While little or no surface deformation has been noted with the isotope irradiation, definite substrate discoloration is observed. This discoloration is much more pronounced when irradiated with a spectrum relatively richer in higher energy particles (Sr90) than when irradiated with a spectrum relatively richer in lower energy particles (Pm147). There is a difference in the way the discoloration appears with polyenergetic irradiation vs. that with monoenergetic irradiation. In the former the discoloration extends more or less uniformly from the irradiated side inwards several mm. In the latter there is little or no discoloration until the "terminal depth" of electron penetration, several mm, is reached.

2.2.2 Irradiation by Dynamitron Accelerator

The irradiation of the GE151 while not as yet complete shows that the deformation of this synthetic fused silica closely parallels that of the previously tested synthetic fused silica, Dynasil.

The fused quartz tested, GE125, shows not only more surface deformation but a curiously mottled discoloration not at all characteristic either in intensity or macroscopic appearance to that seen in Pyrex, BSC2, or synthetic fused silica. These irradication studies are also not complete.

One of the recently developed optical materials of outstanding stability when subjected to thermal shock, Cervit, seems peculiarly sensitive to irradiation as regards surface deformation. A dose as low as 3.5×10^{13} electrons/cm^2 produces a deformation of 3/4 of a fringe (3 times the normal commercial tolerance) whether used as an aluminized substrate or made into a replica diffraction grating. Pyrex, on the other hand, also shows a deformation of about 3/4 of a fringe at 3.5×10^{13} electrons/cm^2 but as the dose is increased, however, the Cervit deforms much more rapidly. At a dose of 2.1×10^{15} the Pyrex shows a deformation of 2.5 fringes while the Cervit shows at least 10 fringes. At this same dose of 2.1×10^{15} electrons/cm^2, BSC2 shows a deformation of about 1 fringe and synthetic fused silica only about 1/3 fringe.

Through the courtesy of the Jarrell-Ash Company we obtained two replica diffraction gratings 2¼" x 2¼" x 3/8" using Vycor as a substrate and were able to include them in a previously planned irradiation. Both gratings showed fringe distortions of about 1/4 fringe before irradiation. One sample was given a dose at 1.0 Mev of 3.5×10^{13} electrons/cm^2 and showed no significant change in deformation. The other received a dose at 1.0 Mev of 2.1×10^{15} electrons/cm^2 and showed a change in deformation of 3 3/4 fringes of 5461A. One curious point of interest regarding the Vycor is that at 3.5×10^{13} electrons/cm^2 the coloration penetration was the 2-3 mm. as normally seen under such irradiation. At the 2.1×10^{15} electrons/cm^2 dose, however, the substrate was browned all the way through to the back, something we have seen in no other material.

2.2.3 Deformation vs. Time Tests

A number of gratings, some irradiated as far back as November of 1966, have been set aside with no special treatment and periodically examined to determine how the surface deformation has varied with time. The data to date is summarized in Table II.

TABLE II

Variation of Radiation Induced Deformation
With Time Since the Irradiation

Deformation (In fringes of 5461(A)

Grating[1]	Pre	Post[2]	Most Recent	Dose(1.0 Mev)
S1B(BSC2)	1/4	1½(9/30/67)	1¼ (8/7/68)	3.5×10^{15}
G2B(BSC2)	1/2	1 (9/30/67)	1/4(8/7/68)	8.4×10^{13}
1231C(Pyrex)	1/8	2½(10/22/66)	1¼ (8/7/68)	$.6 \times 10^{15}$
1251A11(Pyrex)	1/3	1½(10/22/66)	1¼ (8/7/68)	2.8×10^{15}
1251A14(Pyrex)	1/3	1 3/4(10/22/66)	0.9(8/7/68)	3.0×10^{15}

(1) G1B and G2B -- 50 mm. diam. x 10 mm. thick
1231C, 533Q, 1251A11, and 1251A14 -- 2 1/4" x 2 1/4" x 3/8"
(2) Within 48 hours after irradiation

As might be expected, the recovery from the irradiation shows a variation with initial dose and possibly also with the shape of the grating. The most important point, however, is that the gratings do recover and roughly at the rate of 3/4 fringe/year for BSC2 and 3/8 fringe/year for Pyrex.

It will be called that earlier experiments showed that irradiated aluminized substrates when subjected to several hundred degrees centigrade heating did show a recovery to nearly their preirradiation deformation. To this date no means has been found to do this with the replica gratings since they cannot be heated sufficiently without ruining the replication material. Irradiation by intense ultraviolet light has been proposed and an unsuccessful attempt made at relieving the radiation induced stress.

2.2.4 Interferometric Concave Grating Test Bed

While the optics for this unit have been completed, the laser failed during early tests and it was decided to postpone this phase of the project until the forthcoming period. The laser has now been repaired.

2.2.5 Ultra High Vacuum Test Bed

An ultra high vacuum system capable of reaching 2×10^{-8} torr without bakeout and into the 10^{-9} torr range with bakeout has been received. The system is being modified for thermal vacuum stressing of replica gratings at pressures more nearly approximating those of space than we can currently achieve. The initial experiments have involved developing a method of mounting a window of interferometric quality and at the same time preserving the vacuum integrity. The first windows of ordinary 1/2" plate glass tested demonstrated that the proposed O-ring system satisfied the vacuum integrity requirement. One inch thick windows of optical quality are currently being ground and polished.

3.0 Conclusions to Date

Primary effort during this period has involved the testing and modification of the test bed-thermal skid system to allow a wider thermal range through use of a special refrigerant, Dupont E-3. The problems remaining are the procurement of a single pump that will operate over the requisite temperature range and satisfactory insulation of the input brine lines to the test bed.

These thermal vacuum experiments show the importance of the in-situ experiments--experiments that obviously cannot be done at all without the special test bed-thermal skid system designed expressly and uniquely for these measurements. For the first time we are able to duplicate what a replica diffraction grating actually undergoes in a space environment and observe directly how the grating responds under these conditions. The test-stress-test type of tests done earlier were extremely valuable as pilot experiments but their value as predictors of actual in-space flight performance was known even then to be limited.

Irradiation experiments designed to furnish data basic to testing the response of concave replica diffraction gratings have continued. Tests of materials basic to the better replica diffraction gratings have been extended to include fused quartz and Cervit. At this point it would appear that far and away the best material insofar as resistance to deformation by 1.0 Mev electrons is concerned is synthetic fused silica with both Dynasil and GE151 performing substantially the same. It now appears certain that at least for 1.0 Mev electrons that there is no reason why replica diffraction gratings should not be considered as radiation resistant as original masters. This conclusion is reached on the basis of measurements on both replica gratings and aluminized substrates.

4. Plans For Forthcoming Six-Month Period

4.1 Thermal Vacuum Stressing

As soon as the new pump is installed, the thermal vacuum stressing at both high and low temperatures of the rest of the concave gratings will be undertaken. Of particular interest will be the tests of an original master and a replica taken from this master. These vital in-situ measurements are of course possible only through use of the special test bed-thermal skid system designed earlier for this specific set of experiments.

4.2 Particle Irradiation

Primary emphasis will be placed, according to prior HC-NASA conference agreement, on irradiating those concave gratings that pass the thermal-vacuum tests. The value of the previous tests using flat replica gratings and aluminized substrates is now apparent as these tests obviate the necessity of unnecessary testing at radiation doses that are now known to produce no radiation damage. These preliminary tests have deliberately encompassed most of the types of substrate materials used in the manufacture of concave replica diffraction gratings.

4.3 Interferometric Concave Grating Test Bed

With the repair of the laser, work on the interferometric concave grating test bed will be resumed as soon as the test bed-thermal skid system is operational throughout its designed thermal range.

4.4 Ultra High Vacuum Test Bed

The ultra high vacuum system modifications will allow thermal vacuum testing of gratings in the 10^{-8} torr, or better, range. Because of this ultra high vacuum, the system must be much "cleaner" than the other test bed. By design the thermal skid is useable with both systems although not simultaneously. Primary emphasis will be placed on the work outlined in sections 4.1 but present scheduling calls for completion of an optical test unit for use in the ultra high vacuum test bed.

5. Personnel

5.1 Senior Staff

While each of the senior staff participated in some phase of all of the tests, the principal area of responsibility of each investigator is as shown:

Dr. Roy C. Gunter, Jr., Holy Cross College --
principal investigator

Dr. Francis W. Kaseta, Holy Cross College --
vacuum and electrical measurements

5.2 Student Staff

Although many students have been involved in one phase or another of the program, the following were those with specific assignments:

Philip Morrison --
thermal vacuum stressing and line profiles

Michael Kelly --
radiation studies

James Fienup --
ultra high vacuum test bed

5.3 Support from Other Laboratories

Mr. Lester F. Lowe, AFCRL - irradiation tests

Dr. Paul M. Waters, Dr. Richard F. Woodcock, Mr. Samuel F. Walton, Mr. Colin Yates, Research Department of American Optical Co. -- glass stressing and tests

Mr. Richard Schmitt and colleagues of the Jarrell-Ash Co., Grating Laboratories - Replication and tests

Dr. Shields Warren and Mr. Russel Cowing of the New England Deaconess Hospital, Cancer Research

Institute - radiosotope measurements

Dr. Eugene Coyner, E. I. Dupont De Nemours - consultation on wide thermal range refrigerants

Mr. Martin Saepoff, Dynasil, Inc. - samples of Dynasil

Mr. John D. Barker, General Electric, Inc. - samples of GE125 and GE151

Mr. L. M. Donley, Owens-Illinois, Inc. - samples of Cervit.