REPLICA GRATING STUDY
NGR-22-091-002

INTERIM REPORT
July 1, 1967 - December 31, 1967

College of the Holy Cross
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INTERIM REPORT - PHASE III

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Contract No.:	NGR-22-091-002
Date:	February 15, 1968

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Abstract

Thermal-vacuum stressing of concave replica diffraction gratings from three different manufacturers for several days at pressures encountered by orbiting satellites indicates that they will withstand at least temperatures from + 20°C to - 5°C. An irradiated plane replica diffraction grating withstood at least 140°C temperatures for 24 hours at atmospheric pressure with no significant deterioration. Plane replica diffraction gratings that had been previously irradiated withstood temperatures of at least 87°C for 24 hours before failure was detected.

Continuation of the irradiation of plane replica diffraction gratings using BSC2 and synthetic fused silica seems to indicate clearly that at least with electrons of 1.2 Mev energy, the deformation is due to the substrate rather than to the replication process. For the most radiation sensitive material tested thus far, Pyrex, deformations of one-quarter wavelength were not reached until the dose exceeded $6x10^{12}$ electrons/cm^2.

1. Purpose of Effort

1.1 Particle Irradiation

The purpose of the effort in this area during this phase of the investigation was to determine whether indeed the bulk, if not all, of the deformation produced by energetic electron irradiation was due to the substrate rather than the replication. This possibility had been strongly indicated in Phase II.

A secondary effort in this same area was to compare the effects of irradiation during a short time to achieve a given dose as compared with the same dose spread out in a time more closely approximating the true orbital dose time.

1.2 Thermal-Vacuum Stressing

Previous particle irradiation experiments established the validity of approaching the stipulated stress limits through a series of experiments whereby successively greater stress is applied to the gratings rather than hit the gratings with the maximum stress in one experiment. Effort during this period was directed towards the same kind of approach in the area of thermal vacuum stressing.

This course was pursued both as regards the large concave replica gratings and some small expendable plane replica gratings.

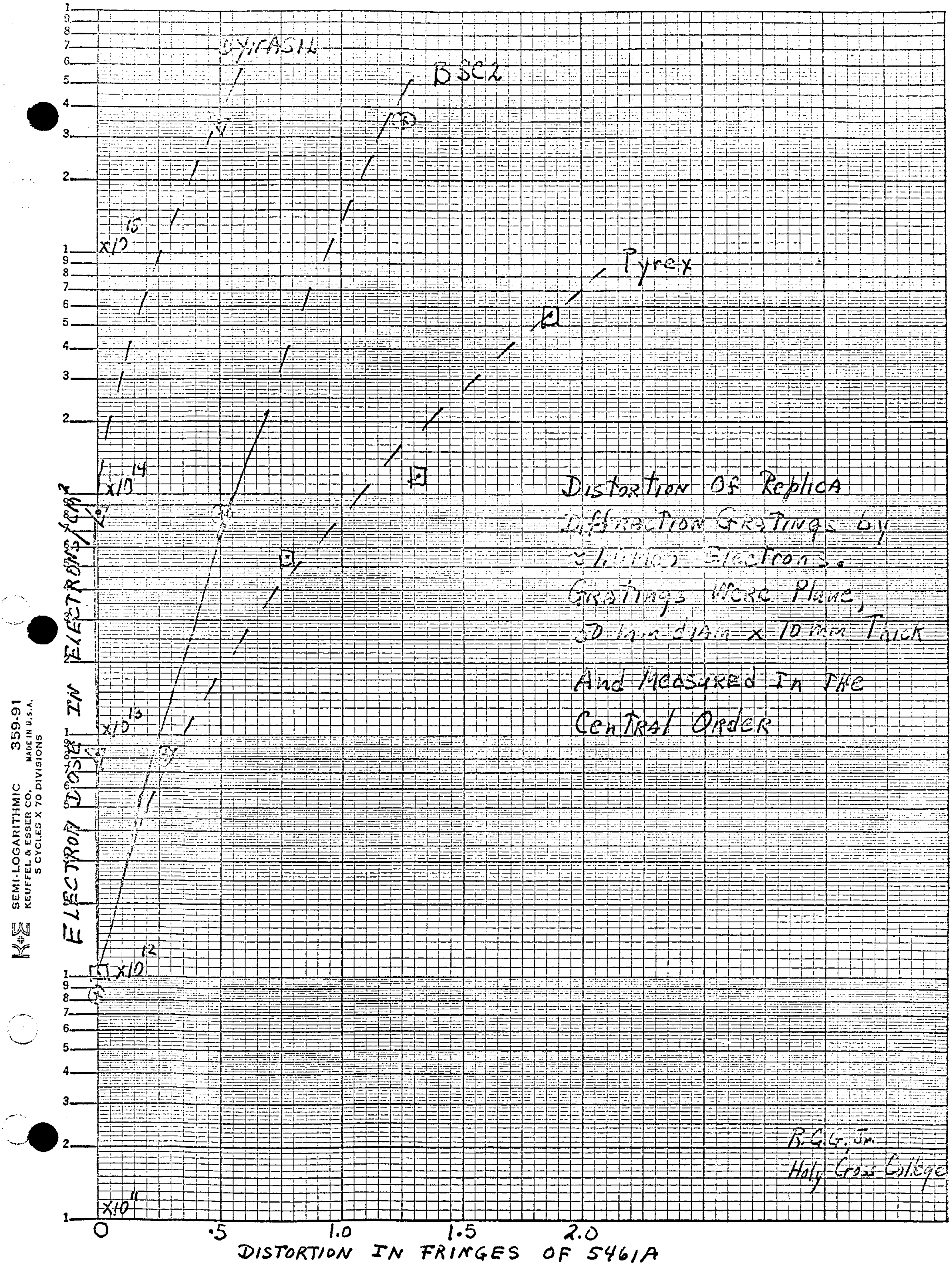
2. Nature of Effort

2.1 Particle Irradiation

2.1.1 Irradiation by Dynamitron

Phase II established the strong likelihood that the deformation observed when replica diffraction gratings were irradiated with 1.0 Mev electrons was actually due to the substrate. The gratings used in Phase II were plane, 50 mm in diameter x 10 mm thickness, and on Pyrex substrates. This work was continued using two other common grating substrates, BSC2 and synthetic fused silica. For these tests our synthetic fused silica was manufactured by the Dynasil Corporation.

The results are shown in the accompanying graph. It should be noted that the original data showed an apparent concave deformation of the Dynasil as compared with the definitely convex deformation of the BSC2 and Pyrex. We feel that this was an error in interpretation of the sign of the Dynasil interferograms prior to irradiation and have so corrected the data to that shown in the graph. The validity of this correction will be studied when some additional synthetic fused silica samples are irradiated.


DYNASIL
BSC2
Pyrex
$\times 10^{15}$
$\times 10^{14}$
$\times 10^{13}$
$\times 10^{12}$
$\times 10^{11}$
ELECTRON DOSE IN ELECTRONS/CM²
0
.5
1.0
1.5
2.0
DISTORTION IN FRINGES OF 5461A
Distortion Of Replica
Diffraction Gratings by
3 Million Electrons.
Gratings Were Plane,
50 mm diam x 10 mm Thick
And Measured In The
Central Order
R.G.G. Jr.
Holy Cross College
SEMI-LOGARITHMIC 359-91
KEUFFEL & ESSER CO. MADE IN U.S.A.
5 CYCLES X 70 DIVISIONS

2.1.2 Irradiation by Radioisotopes

Exposure times for the Dynamitron irradiation were from 5 - 30 minutes. To more closely approximate the orbital dose rates a series of beta emitting radioisotopes was selected with end point energies covering the energy range of interest. The radioisotopes are, Promethium 147, Thallium 204, Strontium 90, and Yttrium 90 (daughter). The concentrations selected are such that it will take approximately six months for the maximal one year equivalent dose of 10^{15} electrons/cm^2 to be accumulated. The gratings will of course be evaluated periodically to study the deformation as a function of dose.

To effect the irradiation the radioisotopes have been evaporated on the bottom of the inside of ointment tins and then sprayed with a very thin layer of plastic which protects the gratings from accidental radioactive contamination. The ointment tins are then placed over a series of replica diffraction gratings <u>and</u> aluminized substrates. Pyrex has been chosen for the substrate as it appears to show the largest deformation. The tests have not started yet as the production of the special 50 mm diam. x 10 mm thick beveled substrates was unavoidably delayed. The irradiation will definitely start in Phase IV.

2.2 Thermal Stressing

2.2.1 At Pressures of 10^{-10} to 10^{-9} torr and Reduced Temperatures.

An experiment was performed at pressures that varied from 10^{-10} to 10^{-9} torr. Concave replica diffraction gratings made by Jarrell Ash, Bausch and Lamb, and Diffraction Products, plus aluminized substrates identical in shape to the gratings were used. The temperature of the gratings as determined by thermocouples epoxied to the mirrors averaged -5°C. The samples were kept under this stress for 4 days. No significant difference in either line profile or dispersion was noticed when spectra taken before and after the thermal vacuum stressing were compared.

It had been planned to go to lower and then higher temperatures but difficulties with the equipment plus higher priority tests precluded these additional tests.

2.2.2 At Atmospheric Pressure

It occurred to us that it would be better to make some preliminary tests to determine just what temperatures the gratings were likely to be able to stand before we "went for broke" and exposed them to the planned thermal extremes. The basic rational was that if we tested the expensive gratings at say 100°C and found them to fail badly, we would never know their actual failure point, information that might be critically valuable had the failure point been, say 95°C. Thus a series of experiments were performed with available plane gratings that had already been used in some earlier particle stress studies. These 50 mm diam. x 10 mm thick gratings and substrates were heated in a laboratory oven at successively higher temperatures. The results are given in Table I.

TABLE I

Thermal Stressing of Replica Gratings at Atmospheric Pressure

Sample*	Previous Irradiation Dose	Dose Energy	Temperature Range	Failure Temperature
G245AP	none	-	60 - 150°C	140°C
G4DA	8.4×10^{11} e/cm^2	1.2 Mev	90 - 110°C	100°C
G6DA	5.0×10^{14} e/cm^2	0.8 Mev	60 - 150°C	87°C
G1DA	3.5×10^{15} e/cm^2	1.2 Mev	90 - 110°C	100°C

* G245AP was on a pyrex substrate, the others were on Dynasil (a synthetic fused silica).

On the basis of this limited sampling, we might conclude for <u>these</u> replica gratings made by JACO that,

1. unirradiated replica gratings will stand upwards of 100°C and possibly as high as 140°C.

2. replica gratings irradiated with 1.2 Mev electrons in doses of 8.4×10^{11} to 3.5×10^{15} electrons/cm^2 withstand up to about 100°C.
3. replica gratings irradiated with 0.8 Mev electrons to a dose of 5.0×10^{14} electrons/cm^2 appear to fail earlier than those irradiated with more energetic electrons but are still good to about 90°C.

Caution should be observed in accepting the above conclusions as final. They are correct for the gratings tested but whether we would get exactly these same results for other samples by the same manufacturer or with gratings produced by other manufacturers, is not at this time known. The results are, however, consistent with known effects of irradiation on plastics and indeed more of the low energy electrons would have been stopped in the plastic than would those of the higher energy. To be on the conservative side we are concluding from these experiments that elevated temperature thermal stressing of the large concave gratings should begin at 70°C, not 100°C.

2.2.3 In-Situ Testing Apparatus

In a series of earlier experiments at Holy Cross plus those at Harvard the desirable characteristics for an HC *in situ* thermal skid were determined. Following these experiments a thermal skid was ordered (to be delivered shortly) with brine ranges of -68 to +21°C with R-22 and R-13 refrigerants or -68 to +138°C with a GE silicone or Dupont Freon E-4 refrigerants. For temperatures above +21°C an electrical heating system will be used with the R-22/R-13 system after the refrigerant has been valved out.

The system will be designed specifically for the type of testing at hand and loads will be limited to about 8 lbs. of glass and aluminum.

2.3 New Grating Test Bed

As had been originally planned, the first work done on the concave gratings was based solely on the variation of line profile and dispersion with thermal vacuum and irradiating particle stress. This was done with a McPherson Model 225 monochromator.

While these tests can be definitive, it was not possible to also measure the efficiency of the gratings, a parameter judged by many to be perhaps the single most important measure of a grating's performance. In our original proposal this necessary weakness of our early work was pointed out but with the subsequent firm commitment by NASA of the necessary funds, a series of design improvement conferences were held with NASA GSFC personnel and later with two independent potential suppliers. Eventually it was decided to continue with McPherson and a new test bed purchased which, when used in conjunction with the original Model 225, would allow us to measure all of the desired parameters.

The test bed was delivered in December and several weeks were then spent in mating it to the Model 225. Since we are after as near diffraction limited performance of the system as we can get, the mating - alignment procedure was of necessity time consuming. This work has now been essentially completed. We are still not entirely satisfied with the performance of the internal photometric system, that system by which the efficiency is measured. To get an independent check on our system, we are having Dr. J. A. R. Samson measure a 1 meter radius concave mirror and are awaiting his results to compare with our own.

2.4 Interferometric Test Bed for Concave Gratings.

It has long been recognized that one of the more powerful tests of the performance of optical components and systems is the interferometer. As has previously been outlined in our reports, we have made extensive use of a Twyman-Green type interferometer for testing plane gratings but felt this type was not as well suited to concave gratings as might be.

We have selected a system used by K.G. Birch[1] of the Light Division, National Physical Laboratory in England for interferometric testing of the concave gratings. Using a He-Ne laser and a set of specially designed external optics we hope to be able to interferometrically examine the gratings while they are mounted in our test-bed. Had there not been a serious delay on the part of the optical component manufacturer, we would have been able to give some actual results. It should be pointed

1. Birch, K.G., "Interferometric Examination Of the Ruling Errors Of A Concave Grating", Jour. Sci. Instr. 43, 243 (April, 1966)

out, however, that prior to ordering the new components a prototype was set up in a two phase program and the results were very encouraging.

3.0 Conclusions to Date

3.1 Effects of Radiation

3.1.1 Irradiation of plane replica gratings made by one manufacturer showed one-quarter wave deformation at exposures of 6×10^{12} e/cm^2 for Pyrex substrates, 1×10^{13} e/cm^2 for BSC2 substrates, and 7×10^{14} e/cm^2 for Dynasil (synthetic fused silica) substrates. The irradiation energy was 1.2 Mev. The deformation increases above these doses.

3.1.2 Irradiation of plane aluminized substrates of Pyrex, BSC2, and Dynasil showed the same variation of surface deformation with irradiation as did the replica gratings.

3.1.3 For the most radiation sensitive material tested thus far, Pyrex, the normal allowable manufacturing tolerance of one-quarter wave is not reached at energies of about 1 Mev until the accumulated dose is 6×10^{12} e/cm^2. This corresponds to a practical upper limit of anticipated dose (see Phase II report, p. 3)

3.1.4 In view of (1) and (2) above we also tentatively conclude that plane replica gratings will probably withstand 1.2 Mev radiation as well as the substrates will alone. The inference, which will be checked later, is that at these energy levels replica gratings will probably withstand as much radiation of this type as will original masters.

3.2 Thermal-Vacuum Stressing

3.2.1 At Orbital Pressure

Experiments conducted in Phases I and II plus those most recently conducted in Phase III with replica gratings from three manufacturers indicate that concave replica gratings will withstand orbital pressures at temperatures of at least +20°C to -5°C. These experiments were generally of several days duration. Conceiveably there may be long term degradations but these have not been indicated so far.

3.2.1 At Atmospheric Pressure

A series of preliminary experiments on plane replica diffraction gratings from one manufacturer indicates that unirradiated replica gratings will withstand upwards of 100°C and perhaps as high as 140°C. The failure of irradiated gratings depends on the previous irradiation history and probably on the nature of the replicating material and the substrate.

4. Plans for Forthcoming Six-Month Period

With delivery of our own thermal skid due within ten days emphasis will be placed during this next period on getting the skid in operation first with a test bell jar and then with our monochromator - test chamber combination. Inasmuch as the bell chamber is not trapped, only second quality gratings will be used in the bell jar. While the thermal skid - bell jar system is being checked out, all gratings will be given their pre-stress analysis (line profile, dispersion, scattered light, and efficiency). After the thermal skid has been checked out with the bell jar, it will be connected into the testbed for the in-situ tests with the good gratings.

The particle irradiation will be limited primarily to stress by the radioisotopes. To get a more quantitative evaluation of the stress, the energy distribution as a function of wavelength as well as the energy at the end point from the radioactive plaques will be determined.

At this point in time it appears unlikely that we will be able to arrive at a testing point where we can irradiate the large concave gratings. This action is in consonance with a decision reached during a conference at GSFC wherein it was agreed that the thermal-vacuum stressing would precede the irradiation of the concave gratings.

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. Edward F. Kennedy, Holy Cross College -- irradiation

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

Prof. Robert F. Kelley, State College at Worcester -- in-situ thermal stressing

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:

John Ebersole -- optical tests

William Mueller and Philip Morrison - line profiles

Mark Roberts and Michael Kelley - radiation studies

5.3 Support from Other Laboratories

Mr. Lester F. Lowe, AFCRL - irradiation tests

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

Dr. E.M. Reeves, Mr. Nathan Hazen, Mr. Frank Kaszinski, Mr. James MacDonald, Harvard College Observatory - thermal-vacuum tests

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

Dr. Irwin Loewen and colleagues of the Bausch and Lomb Co. - Grating Laboratories

Dr. Shields Warren and Mr. Russell Cowing of the New England Deaconess Hospital, Cancer Research Institute - radioisotope plaque preparation and energy distribution measurement.