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REPLICA GRATING STUDY
NGR-22-091-002

FINAL REPORT

June 1, 1966 - April 25, 1975

College of the Holy Cross
Worcester, Massachusetts 01610

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June 1, 1966 - April 25, 1975

Submitted by: College of the Holy Cross
Worcester, Massachusetts 01610

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Contract No.: NGR-22-091-002

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Abstract

This is the Final Report of NASA study contract NGR-22-091-002. The report outlines the methods used to test the response of replica diffraction gratings to a space environment, specifically the response of the replica gratings to thermal-vacuum and electron irradiation stress. It is concluded that there probably is some degradation to thermal stress, but that there is probably no significant degradation due to a vacuum environment. It is further concluded that the degradation of performance of replica gratings because of electron irradiation is due to the interaction of the electrons and the replica grating substrate and not to the replication material itself. Replica and original gratings on the same substrate material should thus respond to particle irradiation in the same manner.

Also included in this report is a study on the variation of index of refraction of a space-related material, $Nd:CaF_2$, with wavelength, percent neodymium doping, and temperature.

1. Purpose of Effort

The primary purpose of this study was to determine whether replica diffraction gratings can be used for vacuum ultraviolet space experiments. Toward this end replica gratings have been given stresses that simulate those found in a one year exposure to a space environment as regards temperature, pressure, and electron irradiation. This report will summarize the results of these experiments. The report will also include the measurement of indices of refraction of a space-related optical material, neodymium doped calcium fluoride.

2. Nature of Effort

2.1 Thermal-Vacuum Stress Experiments

2.1.1 General Methods

The temperature stress experiments were basically divided into two parts. In the first part plane gratings were subjected to pressures down to 10^{-13} torr for 50 hours at 29.0 ± 0.5°C and 11.8 + 0.5°C. Interferometric examination showed no significant (i.e., less than 1/8 λ - λ = 5461A) distortion after this stress. In another series of tests concave replica gratings were tested at ambient (18-26°C) and reduced temperatures (-5°C). The latter samples were held at 10^{-9} to 10^{-10} torr for 4 days. Inspection of the line profiles before and after stressing showed no significant difference. These tests showed that for these gratings over the

range of temperatures and pressures used, there was no significant deterioration after as compared with before thermal-vacuum testing. For details, see Interim Reports on Phases II and III.

In the second part, concave gratings were subjected to in-situ tests in a vacuum monochromator. The pressure was about 10^{-6} torr. These experiments were undertaken to measure what happened to the gratings while they were being thermally stressed. It was planned to do a series of experiments starting in with some old gratings to establish the parametric limitations and to then proceed to the newer gratings furnished by NASA. The following information was evolved (see Phase V Report also);

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 Intensity 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

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 worse 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.

Unfortunately, there developed deficiencies in the thermal skid used in the in-situ experiments and it proved not practical to extend the experiments to the NASA gratings.

2.1.2 General Results and Conclusions

First quality modern replica gratings definitely show no deterioration, as measured in a Twyman-Green interferometer, after thermal-vacuum stress as compared with before thermal-vacuum stress. The question as to whether replica gratings cause deterioration in line beamwidth or efficiency while under thermal-vacuum stress was not as definitively

determined. There is evidence, however, that even old gratings known to be inferior to modern gratings in the stability of the surface do not undergo drastic changes. Less than 10% change was noticed in line width and less than 25% change was noticed in efficiency when grating temperatures were raised from 25°C to 55°C. There was some evidence (see Table I above) that line width tended to worsen as wavelength decreased.

2.2 Particle Irradiation

2.2.1 General Methods

Plane and concave replica gratings were irradiated in air by electrons from a Dynamitron Accelerator. The energy level was 1.0 Mev. The energy dose ranged from about 10^{11} to 10^{16} electrons/cm^2. The dose rate was adjusted in virtually all experiments so that the back of the substrate felt only warm when placed to the cheek immediately after irradiation.

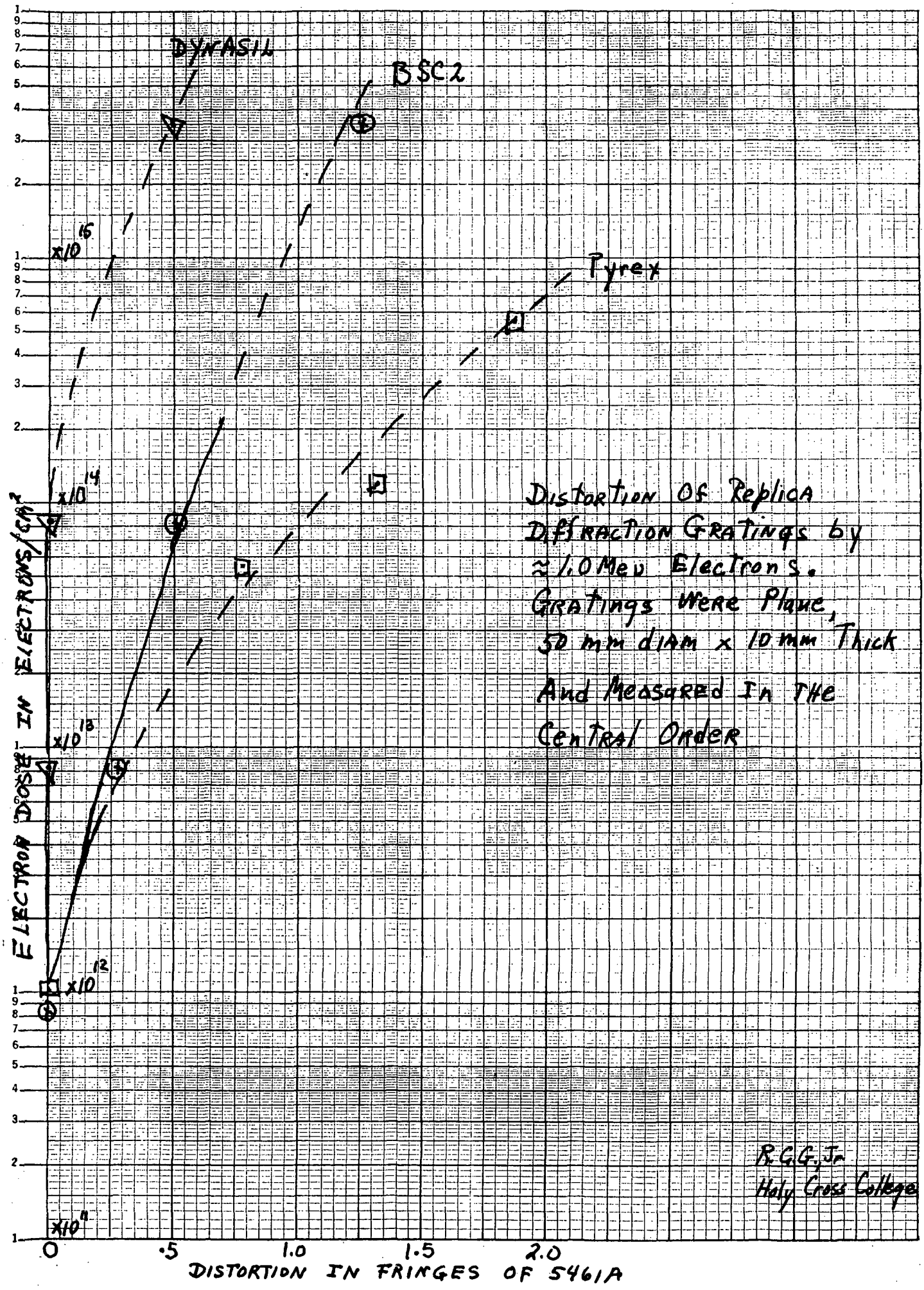
Initial experiments with older replica gratings, mirrors, and substrate material alone indicated that the deformations in the wave fronts diffracted by the gratings were probably due to the substrate rather than the replication process. The deformation was measured by means of a special Twyman-Green interferometer. To quantify this hypothesis plane mirrors and replica gratings with substrates of BSC2, Pyrex, Dynasil (a synthetic fused

silica), GE151 (a synthetic fused silica) GE125 (fused quartz) and Cervit were made. The mirrors and substrates were 50 mm in diameter by 10 mm thick for all samples except the Cervit. In this instance the manufacturer indicated that 8 mm was a sufficient thickness to maintain optical surface figure. These were measured in the Twyman-Green interferometer before and after irradiation.

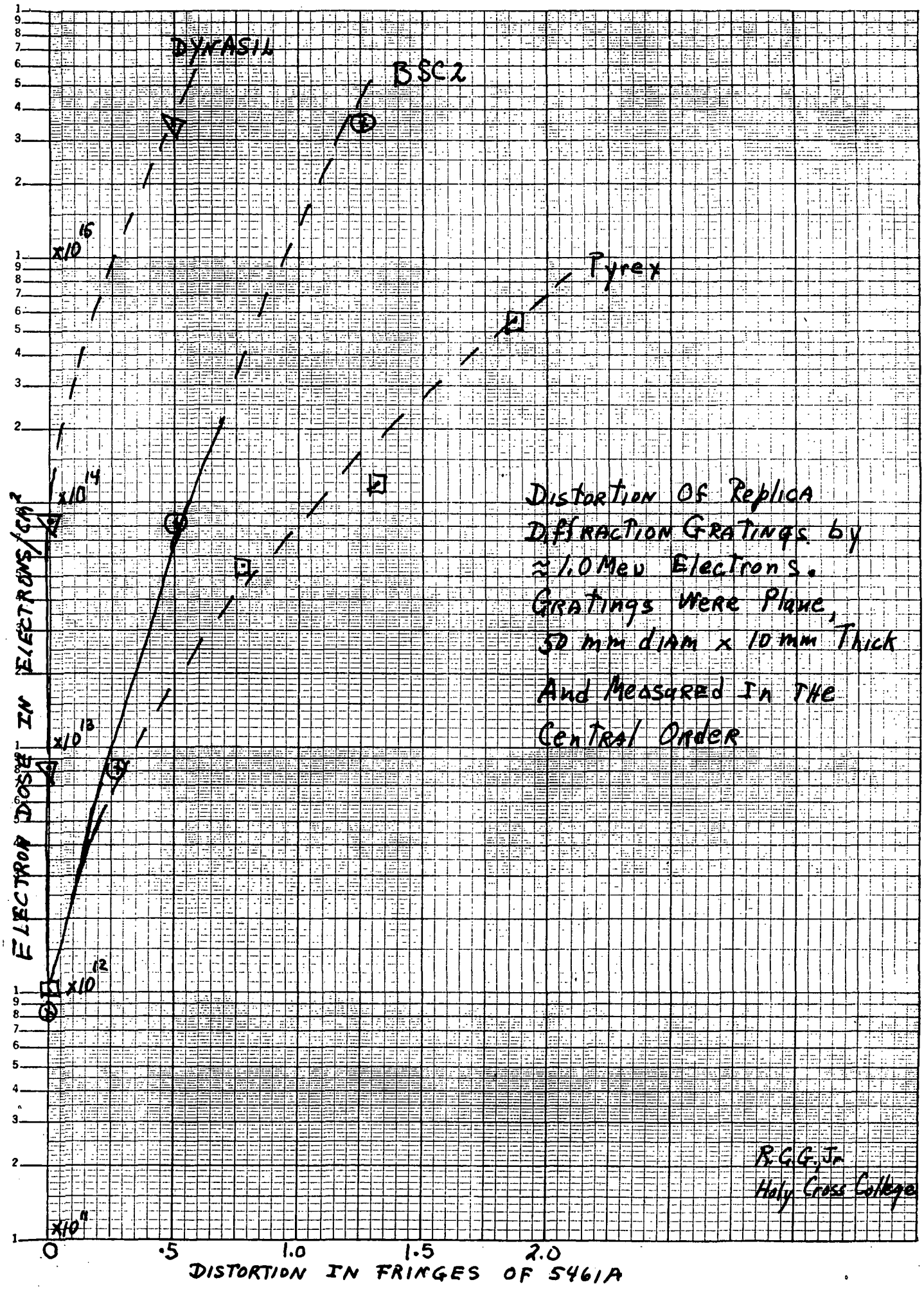
Also irradiated were concave replica gratings made by different manufacturers and on different substrates. These were tested by examining the width and efficiencies of selected spectral lines before and after irradiation in a special monochromator system.

2.2.2 General Results and Conclusions

The results of the tests on the BSC2, Pyrex and Dynasil gratings are shown graphically in Figure 1.



K+E SEMI-LOGARITHMIC 359-91
KEUFFEL & ESSER CO. MADE IN U.S.A.
5 CYCLES X 70 DIVISIONS



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The GE151 results were virtually the same as those for the Dynasil. The GE125 (fused quartz) showed not only considerably more wave front deformation but also a mottled discoloration not at all characteristic in either intensity or macroscopic appearance to that seen in Pyrex, synthetic fused silica, or BSC2. The Cervit gratings seemed particularly 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.

In all cases uncoated substrates and aluminized substrates showed the same (within $\pm$ 1/8 λ) deformations as those replica diffraction gratings made using the same material as a substrate. The hypothesis that the deformation observed is due to the substrate is thus on a firm footing.

In our irradiation studies of the concave replica diffraction gratings, a good degree of consistency of results was found with respect to substrate types. Manufacturer inconsistencies can thus be ignored. In other words, Pyrex gratings, as an example, all performed similarly regardless of manufacturer. Also, even though the irradiated gratings were actually hotter after the 3.6×10^{14} e^-/cm^2 irradiation than after the 10^{15} level, and the more severe grating deterioration was after the latter dosage, thermal effects of the irradiation were probably less important than the irradiation itself. Hence the poor performance of the BSC-2 substrates after intense irradiation is significant. Pyrex gratings seemed reasonably stable from our tests, while the Dynasil actually seemed to improve. A summary of selected grating data after Dynamitron irradiation is shown in Table II.

TABLE II

(Selected Data for 2945Å Line of Helium)

Grating Substrate*	Irradiation Level ($\times 3.6e^{-}/cm^2$)	Half-Power Beamwidth (A)	Tenth-Power Beamwidth (A)	Efficiency (%)
Pyrex-D	0	0.300	0.660	1.76
	10^{13}	0.250	0.615	1.80
	10^{14}	0.262	0.548	1.65
	10^{15}	0.288	gross	--
Pyrex-J	0	0.200	0.380	32.0
	10^{13}	0.245	0.502	35.6
	10^{14}	0.242	0.508	30.0
	10^{15}	0.272	0.612	29.0
Dynasil	0	0.305	0.560	--
	10^{13}	0.325	0.588	32.1
	10^{14}	0.270	0.477	25.2
	10^{15}	0.230	0.465	23.4
BSC2-B	0	0.270	0.560	36.0
	10^{13}	0.246	0.490	28.0
	10^{14}	0.234	0.446	32.0
	10^{15}	gross	gross	--
BSC2-J	0	0.130	0.305	38.9
	10^{13}	0.175	0.385	32.0
	10^{14}	0.238	0.427	27.5
	10^{15}	$>$ 0.800	gross	--

*Suffixed letter indicates manufacturer

It is concluded on the basis of the studies of uncoated and aluminized substrates, plus replicated diffraction gratings on the same substrate material, that it is the interaction of the electrons with the substrate that causes degradation of performance. The reason is that electrons of the energy level tested, 1.0 Mev, pass through the aluminum and replicating base and terminate in the substrate. On this basis it is most probable that original gratings will respond the same as replicated gratings to high energy electrons.

2.3 Refractive Index of Nd: CaF_2 and Some Nd Doped Glasses as a Function of Wavelength, % Neodymium, and Temperature

An ancillary series of experiments was carried out to determine the variation of the index of refraction of Nd:CaF_2 and some Nd doped glasses as a function of wavelength, % Neodymium, and temperature.

2.3.1 General Methods

The measurements were made on a Bausch and Lomb Precision Refractometer. A special housing was fabricated to keep the sample at the desired temperature. The samples were in the form of rectangular parallelepipeds. The surface of the sample that contacted the working prism of the refractometer was ground and polished flat to $\pm \lambda/8$ of 5461A radiation. The basic

calcium fluoride was 99.999% pure. The purity of the rare earth dopant was 99.99% or better.

2.3.2 General Results and Conclusions

The results of the study are summarized in Tables III - IV. This work was published in APPLIED OPTICS, Vol. 14, No. 1, 174, January 1975.

TABLE III

Composition of Neodymium Doped Glasses in Weight Percent

Nd_2O_3	SiO_2	K_2O	Rb_2O	BaO	Sb_2O_3
0.5	73.5	10	10	5	1
1	73	10	10	5	1
2.55	71.45	10	10	5	1
3	71	10	10	5	1
15	59	10	10	5	1

TABLE IV

Refractive Index of $Nd:CaF_2$ at 25.0°C ± 0.2°C

Å	%Nd	0.001	0.01	0.1	0.5	1	10
6678.15	(He)	1.43225	1.43233	1.43271	1.43339	1.43588	1.45556
5438.47	(Cd)	---	1.4328	---	---	1.4364	---
5892.90	(Na)	1.43379	1.43385	1.43426	1.43495	1.43744	1.45738
5875.87	(He)	1.43382	1.43387	1.43432	1.43500	1.43749	1.45739
5790.65	(Hg)	1.43402	1.43408	1.43451	1.43519	1.4376	---
5769.59	(Hg)	1.43404	1.43412	1.4346	1.4352	---	---
5460.74	(Hg)	1.43491	1.43499	1.43537	1.43610	1.43856	1.45854
5350.46	(Tl)	1.43520	1.43521	1.43572	1.43643	1.43888	1.45892
5085.82	(Cd)	1.43608	1.43615	1.43657	1.43729	1.43971	---
5015.68	(He)	1.43635	1.43644	1.43687	1.43753	1.44000	1.46011
4921.93	(He)	1.4367	---	---	---	---	---
4799.92	(Cd)	1.43718	1.43726	1.43770	1.43839	1.44079	1.46107
4713.37	(He)	1.4376	---	---	---	---	---
4678.16	(Cd)	1.4377	---	---	---	---	---
4471.48	(He)	1.43879	1.43880	1.43930	1.44000	1.44236	1.46274
4358.35	(Hg)	1.43944	1.43953	1.43994	1.44063	1.44308	1.46350

TABLE V

Change in Refractive Index with % Neodymium

Wavelength Å	$10^3 \times (dn)/(dc_{Nd})$	Correlation coefficient
6678.15	2.309	0.9985
5892.90	2.337	0.9985
5875.87	2.335	0.9985
5790.65	3.373	0.9812
5769.59	2.266	0.9716
5460.74	2.341	0.9985
5350.46	2.374	0.9986
5085.82	3.434	0.9820
5015.68	2.352	0.9986
4799.92	2.366	0.9987
4471.48	2.374	0.9988
4358.35	2.384	0.9987

TABLE VI

Refractive Index of Nd:Glass at 25.0°C ± 0.2°C

Å	%Nd	0.5	1	2.55	3	15
6678.15	(He)	1.4947	1.4955	1.4989	1.5009	1.5345
6438.47	(Cd)	1.4954	1.4963	1.4996	1.5016	1.5354
5892.90	(Na)	1.4975	1.4983	1.5020	1.5037	1.5362
5875.87	(He)	1.4976	1.4984	1.5021	1.5038	---
5769.59	(Hg)	1.4979	1.4988	1.5024	1.5041	---
5460.74	(Hg)	1.4995	1.5004	1.5046	1.5058	1.5400
5350.46	(Tl)	1.5001	1.5009	1.5043	1.5063	1.5401
5085.82	(Cd)	1.5016	1.5026	1.5059	1.5080	1.5425
5015.68	(He)	1.5021	1.5029	1.5064	1.5084	1.5430
4921.93	(He)	1.5027	1.5036	---	1.5091	1.5437
4799.92	(Cd)	1.5036	1.5045	1.5079	1.5100	1.5448
4713.37	(He)	1.5042	---	---	1.5106	---
4678.16	(Cd)	1.5046	1.5055	1.5088	1.5110	1.5459
4471.48	(He)	1.5064	1.5073	1.5107	1.5129	1.5480
4358.35	(Hg)	1.5076	1.5085	1.5122	1.5141	1.5494
4046.56	(Hg)	1.5113	---	---	---	---

TABLE VII

Refractive Index of $Nd:CaF_2$ as a Function of Temperature

Wavelength Å	Temperature °C	0.001	0.01	0.1	0.5	1	10
6678.15	15	1.43234	1.43244	1.43286	1.43349	1.43601	1.45571
	25	1.43225	1.43233	1.43271	1.43339	1.43588	1.45556
	35	1.43215	1.43228	1.43263	1.43334	1.43583	1.45550
	55	1.43197	1.43107	1.43241	1.43316	1.43560	1.45524
5875.87	24.3	1.43882	1.43386	1.43432	1.43499	1.43749	1.45738
	36.8	1.43369	1.43375	1.43416	1.43491	1.43739	1.45725
	58	1.43347	1.43353	1.43397	1.43466	1.43715	1.45696
	67.9	1.43341	1.43349	1.43389	1.43467	---	1.45691
5460.74	15	1.43502	1.43511	1.43548	1.43621	1.43872	1.45868
	25	1.43491	1.43499	1.43537	1.43610	1.43856	1.45854
	35	1.43476	1.43494	1.43525	1.43604	1.43849	1.45846
	55	1.43455	1.43473	1.43503	1.43582	---	1.45819
4358.35	15	1.43960	1.43964	1.44013	1.44078	1.44326	1.46369
	25	1.43944	1.43953	1.43994	1.44063	1.44308	1.46350
	35	1.43937	1.43942	1.43983	1.44049	1.44300	1.46335
	55	1.43919	1.43924	1.43973	1.44031	---	1.46320

3. Personnel

3.1 Senior Staff

Dr. Roy C. Gunter, Jr. --- principal investigator

From time to time various other senior staff members worked on the project. All are referenced in the Interim Reports covering the period and nature of the particular staff member's effort.

3.2 Student Staff

A considerable number of students were involved in this project. The names of the students and the nature of their work is referenced in the Interim Reports. The only exception is that of Joseph V. Closs who made the measurements of refractive index following issuance of Interim Report - Phase VII.

3.3 Support from Other Laboratories

This project was extremely fortunate in the support given it by grating and other optical manufacturers, plus that from various government laboratories. The particular contribution of each is acknowledged in the appropriate Interim Reports. Exceptions are those involved with the last work on the project, viz. the refractive index measurements. Here we would like to thank Walter Hargraves of Optovac, Inc., who supplied the calcium fluoride, and

William Prindle of the American Optical Company, who supplied the neodymium doped glass. Irving H. Malitson and Marilyn J. Dodge of the National Bureau of Standards furnished us with six place measurements of the refractive indices of several of our own glasses. Given W. Cleek and Roy M. Waxler of the National Bureau of Standards were very helpful in supplying us with samples from melts of fused borate glass (glass E1583) that had been accurately measured, particularly as regards the change in refractive index with temperature at the National Bureau of Standards.

We would also like to thank Colin Yates and Raoul Boulanger of the American Optical Company Precision Glass Shop for aid and advice on the grinding and polishing of the samples. Finally, we acknowledge with great gratitude the assistance given by James J. Chisolm and G.B. Coniglio of the Bausch & Lomb Optical Company in setting up special tables for our refractometer as a function of wavelength and temperature.

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