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In-House Report
July 1993

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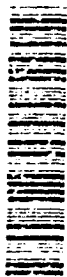


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THE PERFORMANCE AND SURVIVABILITY OF DICHROMATED POLY (VINYL ALCOHOL) HOLOGRAMS FOR SPACE BASED PHOTONIC APPLICATIONS

Steven P. Hotaling, Rome Laboratory; Gurusamy Manivannan,
Rupak Changkakoti, Roger A. Lessard, Quebec, Canada

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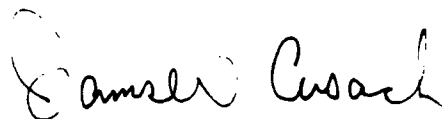
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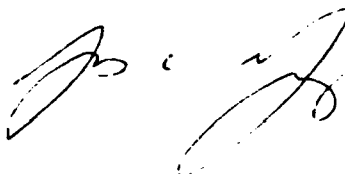
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1. AGENCY USE ONLY (Leave Blank)		2. REPORT DATE July 1993		3. REPORT TYPE AND DATES COVERED In-House Nov 91 - Mar 92	
4. TITLE AND SUBTITLE THE PERFORMANCE AND SURVIVABILITY OF DICHROMATED POLY (VINYL ALCOHOL) HOLOGRAMS FOR SPACE BASED PHOTONIC APPLICATIONS				5. FUNDING NUMBERS PE - 62702F PR - 4600 TA - P1 WU - 11	
6. AUTHOR(S) Steven P. Hotaling, Rome Laboratory; Gurusamy Manivannan, Rupak Changkakoti, Roger A. Lessard, Quebec, Canada					
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Rome Laboratory (OCPA) Centre d' Optique 25 Electronic Parkway Photonique et Laser (COPL) Griffiss AFB NY 13441-4515 Pavillon A.-Vachon (see reverse)				8. PERFORMING ORGANIZATION REPORT NUMBER RL-TR-93-131	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Rome Laboratory (OCPA) 25 Electronic Parkway Griffiss AFB NY 13441-4515				10. SPONSORING/MONITORING AGENCY REPORT NUMBER	
11. SUPPLEMENTARY NOTES Rome Laboratory Project Engineer: Steven P. Hotaling/OCPA (315) 330-3147					
12a. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution unlimited.				12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) The results of an experiment which subjected high diffraction efficiency holograms (average: 40%, max. = 58%) written into DCPVA (Dichromated polyvinyl alcohol) photopolymer thin films to the combination of ionizing radiation, temperature and atomic oxygen. This simulated to some extent, the synergistic effects of the LEO (Low Earth Orbital) environment. Remarkably, diffraction efficiency was not found to be significantly degraded in these experiments. A regression model for gamma ray radiation induced degradation in DCPVA holograms is presented.					
14. SUBJECT TERMS photopolymer, dichromated poly (vinyl alcohol), photonics, photonic memories, hologram				15. NUMBER OF PAGES 20	
				16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT UNCLASSIFIED	18. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED	19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED	20. LIMITATION OF ABSTRACT U/L		

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The Performance and Survivability of dichromated poly(vinyl alcohol) Holograms for Space Based Photonic Applications

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The results of an experiment which subjected high diffraction efficiency holograms (average: 40%, max.= 58%) written into DCPVA (Dichromated polyvinyl alcohol) photopolymer thin films to the combination of ionizing radiation, temperature and atomic oxygen. This simulated to some extent, the synergistic effects of the LEO (Low Earth Orbital) environment. Remarkably, diffraction efficiency was not found to be significantly degraded in these experiments. A regression model for gamma ray radiation induced degradation in DCPVA holograms is presented.

I. Introduction

Optical interconnects, mirrors, lenses, prisms, and other optics comprising photonic breadboard systems occupying large, massive optical tables today must be miniaturized and fabricated into light weight engineering packages for aircraft applications. For weight critical satellite applications, the synergistic effects of ionizing radiation, reactive plasmas, contamination, and temperature present additional concern.¹⁻³

Photopolymer Holographic Optical Elements (HOEs) are capable of replacing conventional optical devices providing drastic space and weight reduction^{5,6,7}. Moreover, the mechanical versatility of photopolymers (ability to coat complex shapes such as aspherical surfaces) offers flexibility to systems design engineers. Conventional belief however, is that photopolymers and photo-crosslinking materials could not survive the above synergistic effects of the space environment with sufficient reliability throughout the mission lifetime.

This hypothesis was examined in this experiment by subjecting several (72) high diffraction efficiency holograms written into DCPVA to varied doses of gamma ray radiation (0, 2.5 and 10 Mrads) in an ozone environment at 83 degrees centigrade. The characteristics of the sample populations are given in section 2. The gamma source used was a Co⁶⁰ source with a dose rate of 2 Mrad/hour. This total radiation dose, ozone density and thermal environment is a reasonable worst case operational scenario for simulation of accelerated life testing of enclosed photonic devices in a class of LEO-tasked satellites.¹⁻³

It should be noted of course, that this radiation dose rate is much too high for the natural space radiation environment and thermal condition. Such conditions, persisting on a satellite, would represent a worst-case sun-facing bulkhead with faulty thermal control unit. The ozone environment is similarly exaggerated relative to the actual mission environment. This experimental environment is then, a form of accelerated life cycle test for space based DCPVA photonic devices.-

2. Experimental

2.1 Experiment Design:

The experiment was designed to characterize the loss in DCPVA HOE diffraction efficiency due to radiation treatment

Note that the synergistic effects of radiation, oxygen plasma, and temperature during the radiation procedure are grouped into the term radiation treatment.

The procedure followed to test these hypotheses was:

- (A) Prepare several DCPVA plates and write "identical" point holograms per plate
- (B) Thermo-chemically fix half of the samples, leaving the others in the intrinsic state
- (C) Measure the Diffraction Efficiency of each point hologram
- (D) Radiation Treat both sets of samples
- (E) Measure the diffraction efficiency
- (F) Model Results

There were three identical sets of plates, each containing 24 samples divided into three experimental classes of 8 samples: Intrinsic, Thermo-Chem, and Radiated. The intrinsic class contained DCPVA holograms written into DCPVA and left "unfixed" or undeveloped. The thermo-Chem class contained holograms "fixed" or developed by thermo-chemical processing. The Radiated class was treated with 0, 2.5 and 10 Mrads of gamma rays while in an ozone environment at 83°C.

2.2 DCPVA Film Preparation

The DCPVA samples were prepared* as follows:

1. The PVA dry powder ($\bar{M}_n = 11 \pm 31000$; Aldrich 100% hydrolyzed) was dissolved in doubly distilled water in a heated, agitated water bath. The solution was held at 80°C. A 7 weight percent PVA aqueous solution was obtained. To this, a desired amount of ammonium dichromate solid was added to the polymer solution to obtain a 1.2 wt.% ammonium dichromate aqueous polymer solution which had natural pH.
2. Thick films of the DCPVA solution were obtained by pipetting 4ml of the photopolymer solution onto previously cleaned (with chromic acid and DI and oven dried

prior to coating) glass microscope slides placed upon a leveled fume hood bench. The slides were allowed to dry for 24 hours, resulting in polymer films with thicknesses of 55-60 microns.

2.3 Hologram Preparation

1. The plates were exposed to a laser interference pattern formed by focusing two laser beams at a point; generating a point hologram⁸ as shown in Figure 1. The two recording beams emanated from a Spectra Physics Argon Ion laser (wavelength = 488 nm), and the real time generation of the holograms was monitored with the 647.1 nm beam from a Spectra Physics Krypton laser. The higher energy 488nm optical excitation energy causes cross linking in DCPVA films due to the electron transfer reaction involving Cr(VI) and the PVA matrix, leading to the formation of Cr(III) as the photoproduct. This mechanism, illustrated in Figure 2, was verified by detailed electron paramagnetic resonance (EPR) spectroscopy experiments¹⁰.

Viewed in white light, the holograms appeared as interference patterns as shown in Figure 3. Figure 3 is a double exposure photograph which shows another reading beam (HeNe laser wavelength = 632.8 nm) superimposed upon the plate. The reconstructed images obtained were those expected for a point interference pattern. A typical reconstructed image is shown in Figure 4. The read beam was aligned to the Bragg angle. All holograms were recorded with an interbeam angle of 40 degrees (i.e. 1401 cycles/mm) and a beam intensity ratio of unity. The intensity of the laser beam was measured using a Radiometer/Photometer supplied by EG&G Gamma Scientific (Model 550-1).

The real time diffraction efficiency is defined as :

$$\eta = I_1/I_t$$

Where I_1 is the intensity of the first order diffracted beam and I_t is the intensity of the transmitted beam at normal incidence through the unexposed film.

2. Thermo-chemically fix a subpopulation of the hologram plates by heating in a 20% water and 80% ethanol agitated water bath at 85°C¹¹. This is followed by soaking for three minutes in a 100% ethanol solution. The holographic plates were then dried with high pressure air.

The relative frequency distributions of the diffraction efficiencies for the Thermochemically fixed and un-fixed holograms appear as shown in Figures 5 and 6, respectively. As noted in the literature, thermochemical fixing of the holograms increases the diffraction efficiency by 10 to 30 percent (depending upon precursor polymer film preparation parameters such as pH, and molecular concentrations)^{8,9}.

2.4 Gamma Ray Irradiation

The photopolymer plates and blank glass substrates were irradiated in a Co⁶⁰ source designed to yield a symmetric gamma ray flux. This symmetry results from a source with 12 source-containing rods forming a cylinder into the center of which the samples were

located during irradiation. The source was operated by Rome Laboratory Materials Science Directorate (Hanscom AFB). The dose rate was 2Mrad per hour at a temperature of 83°C. The radiation dose was measured by a calorimeter. The chamber had an ozone flux of 0.1 cm⁻³. The DCPVA films did not peel from the substrate due to radiation treatment and the surface of the DCPVA polymer also did not degrade due to this environment.

The diffraction efficiency of the irradiated holograms is given as the relative frequency distributions of Figure 7 and 8. Figures 7 and 8 correspond to the holographic plates, which were treated with 2.5 and 10 Mrads of gamma rays, respectively. Notice that the percentage difference in diffraction efficiency between the Fixed and unfixed plates is very small. This implies that the state of development of the hologram is not an issue for gamma ray irradiation for these dose rate and total flux levels. The diffraction efficiency measurements were carefully performed to account for photodarkening of the substrate versus the degradation of the photopolymer. Analysis of the variance of these data provided the following regression model for the data:

$$\eta = 0.84 - 4.23e^{-2\Phi}$$

Where Φ is the total gamma ray dose.

3. Conclusions

The DCPVA photopolymer was found to be remarkably resistant up to the investigated 10 Mrads of gamma ray radiation at 80°C, while in an ozone environment. This environmental condition is similar to that expected to exist in sealed spacecraft photonics modules after mission completion. These results suggest that more research should be performed on photopolymer radiation effects to determine HOE failure thresholds; determining the potential photopolymer spacecraft applicability. Also encouraging is the result that the surface was not degraded by the ozone flux in the chamber. These data suggest that further work be performed on DCPVA photopolymer transmission and reflection holograms for space based systems applicability.

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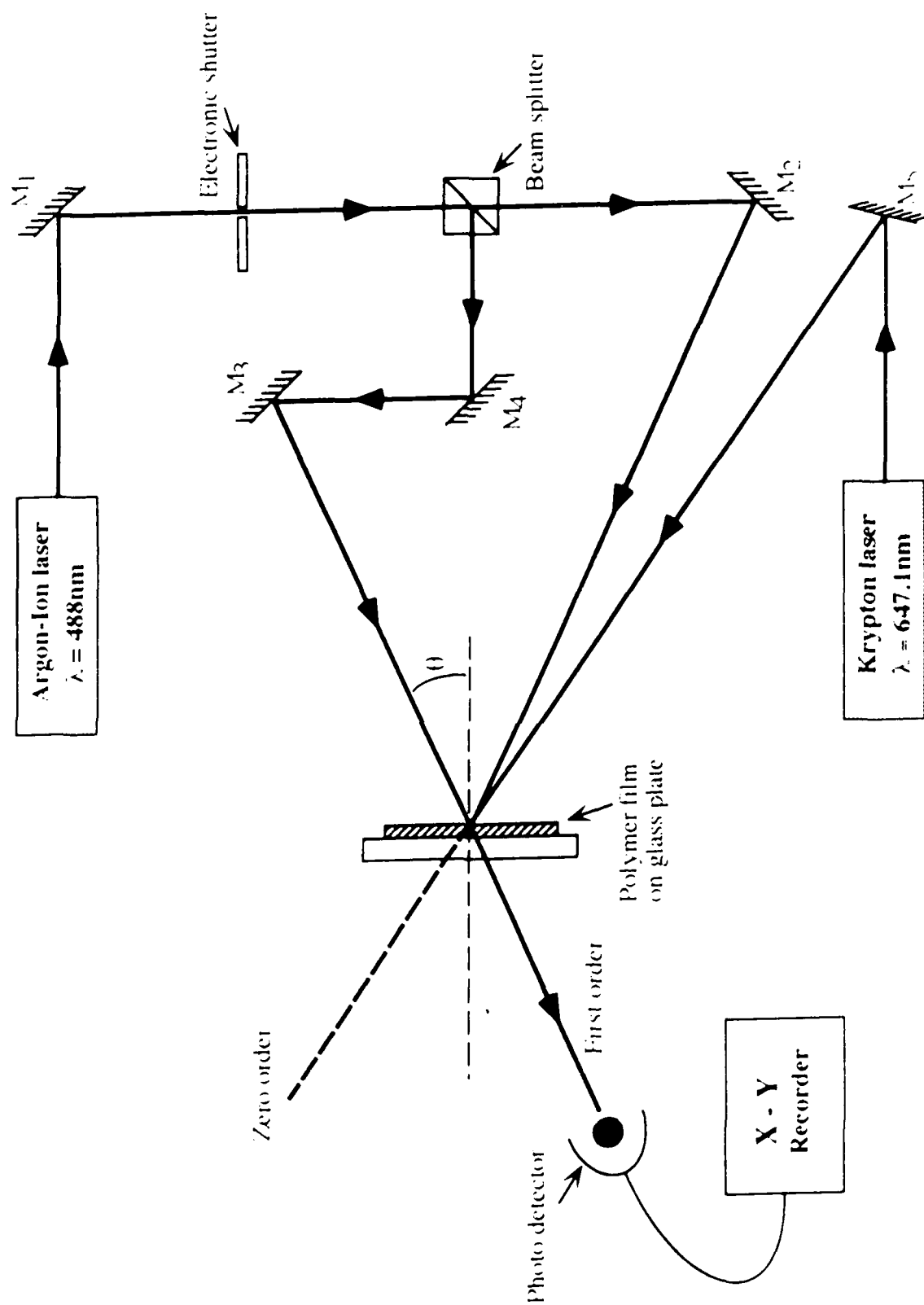


Figure 1. Point hologram read/write geometry.

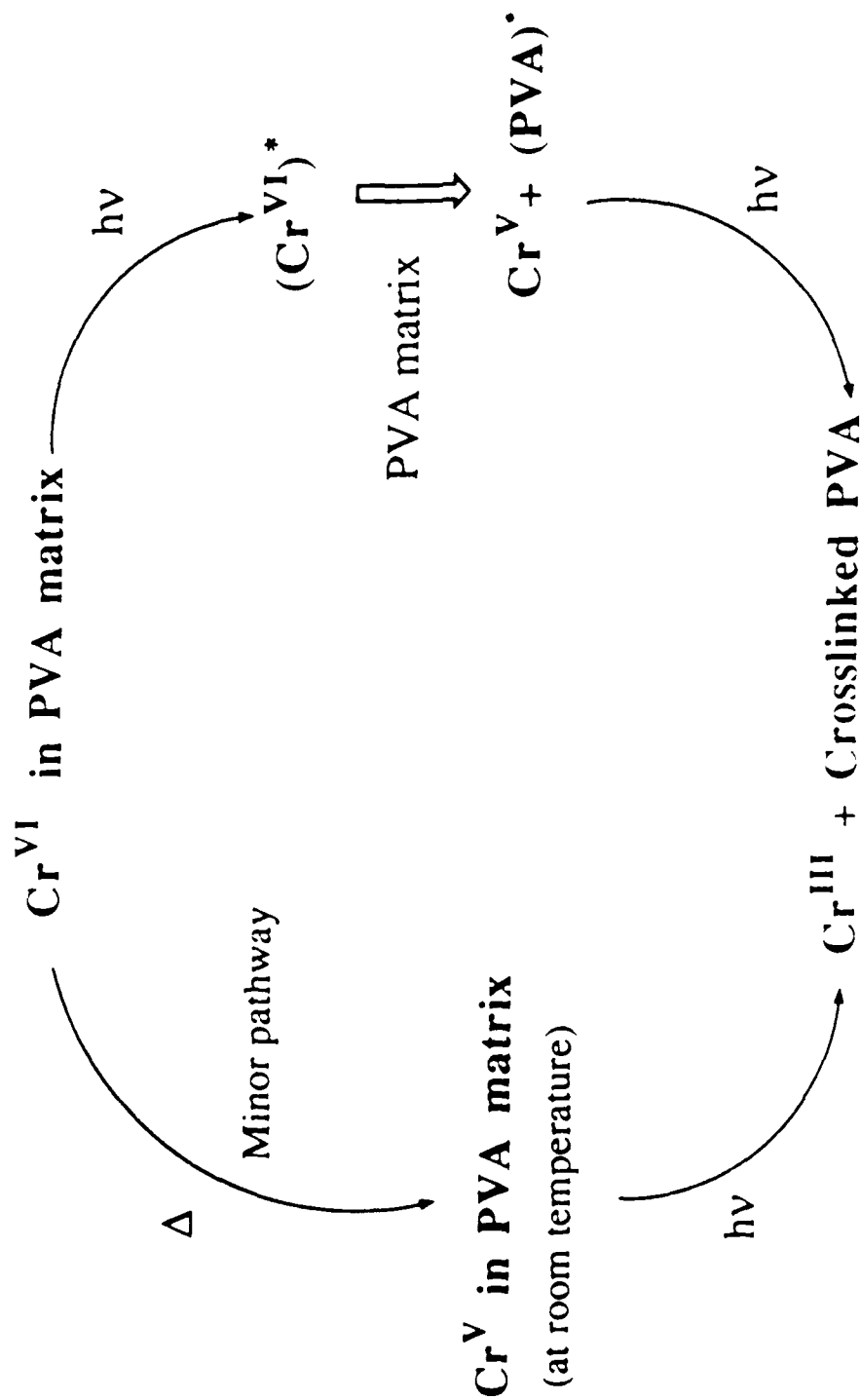


Figure 2. The reaction dynamics associated with DCPVA hologram formation. Upon optical excitation, $\text{Cr}(\text{VI})$ in the polymer matrix undergoes electron transfer producing $\text{Cr}(\text{V})$ and polymer radical. The $\text{Cr}(\text{V})$ undergoes further reaction generating the photoproduct $\text{Cr}(\text{III})$ and crosslinked polymer.

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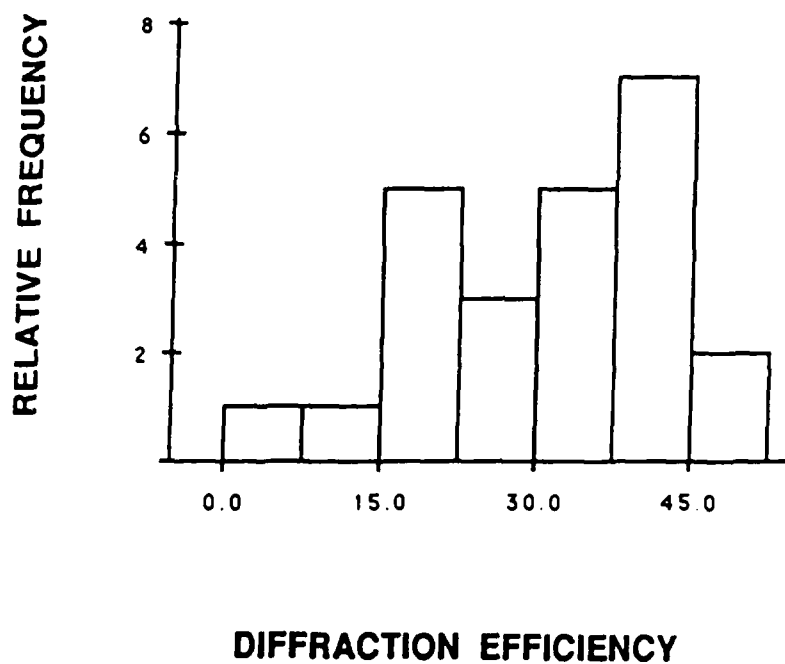


Figure 5. Relative Frequency Distribution of the diffraction efficiency of the thermochemically fixed DCPVA holograms.

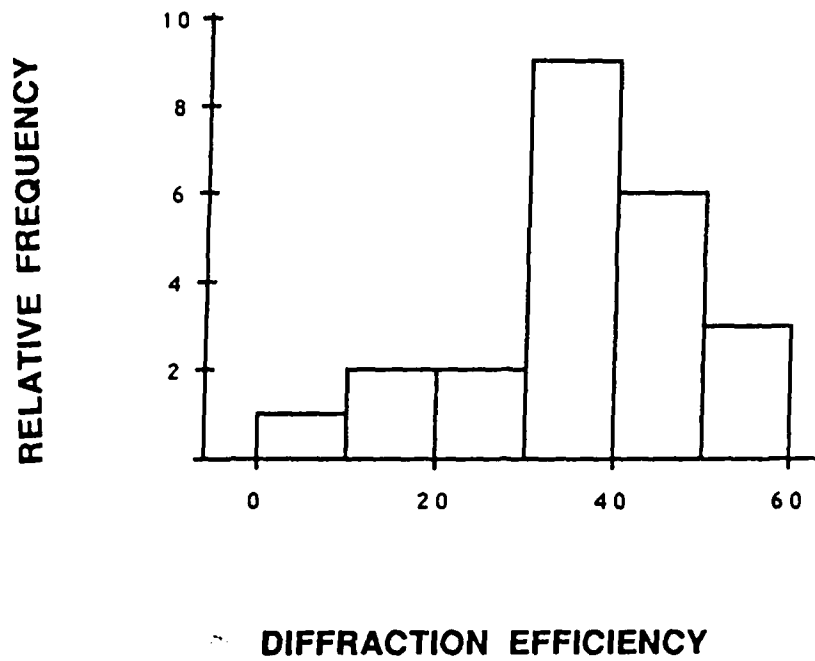


Figure 6. Relative Frequency Distribution of the diffraction efficiency of the unfixed DCPVA holograms.

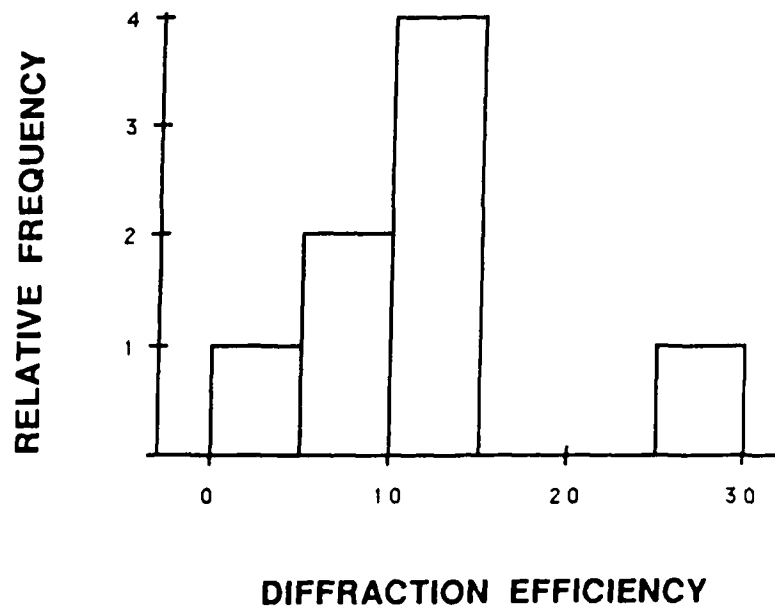


Figure 7. Relative Frequency Distribution of the diffraction efficiency of the DCPVA holograms after treatment with 2.5 Mrads of Gamma ray radiation.

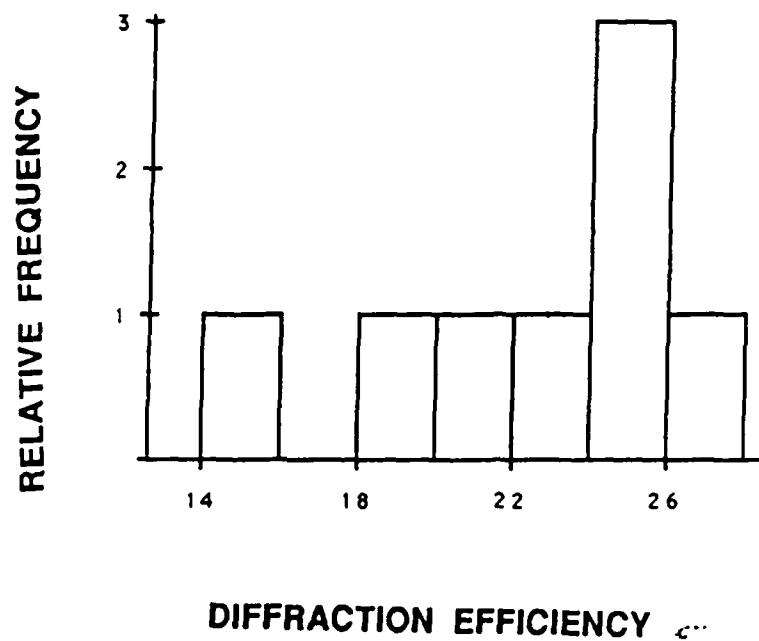


Figure 8. Relative Frequency Distribution of the diffraction efficiency of the DCPVA holograms after treatment with 10 Mrads of Gamma ray radiation.

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