New recording materials for holography

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Abstract

Some of the alternative silver halide materials that can be considered to replace the previous Agfa Holotest materials are discussed. The characteristics of Slavich and HRT materials are examined, including the special aspects of recording and processing ultrahigh-resolution silver halide materials. These materials can offer a significant improvement in product quality. The sensitivity is lower than that of Agfa materials but the signal-to-noise ratio and dynamic range is an improvement. Photopolymers are increasingly being used for mass production. Dupont's OmniDex material, combined with their colour tuning film is an excellent material for this purpose as no wet processing is involved.

1. Background

The fact that AGFA holographic materials will no longer be produced is of great concern to many companies and individuals in holography. However, the *MILLIMASK* plates (green sensitive) high-resolution materials will still be manufactured by AGFA. This product can replace the former AGFA *HOLOTEST 8E56* holographic plates. There are alternative silver-halide materials which in some cases are not only a replacement but, in addition, can offer a significant improvement in product quality, e.g., masters for display holography and some HOEs. The sensitivity is lower compared with AGFA materials, but smaller grain sizes offer a higher signal-to-noise ratio and dynamic range. Masters and HOEs recorded on ultra-high resolution silver-halide emulsions are similar to DCG holograms and suitable for mastering. It is possible to hypersensitise such emulsions in order to increase their sensitivity. (Ref. 1, Chapter 6)

Mass production of holograms on film has more or less already been shifted towards *photopolymer* materials. E.I du Pont de Neumours & Co. has a monochrome product on the market, the *OmniDex* material. This product combined with the colour-tuning film is an excellent material for mass production since no wet processing is required, only UV-curing and baking are needed. Later this year DuPont will introduce a panchromatic photopolymer material for colour holography.

Primarily, the main problem is to find alternatives for pulsed ruby-laser recorded holograms (wavelength 694 nm) as well as large-format (square-meter size) film holograms (very large-format Benton transmission and Denisyuk reflection display holograms).

2. Silver-Halide Recording Materials

2.1 EMULSIONS

2.1.1 Introduction

A *silver-halide recording photographic material* is based on one type, or a combination of silver-halide crystals embedded in a gelatin layer, commonly known as the *photographic emulsion.* Actually, the photosensitive emulsion is not really an "emulsion" but rather a thin film of silver-halide microcrystals dispersed in a colloid (gelatin). However, the term "emulsion," is commonly used in photography for this perpetual suspension. The emulsion is coated on a flexible or stable substrate material. There are three types of silver-halides: *silver chloride* (AgCl), *silver bromide* (AgBr), and *silver iodide* (AgI). Silver chloride is used for low sensitivity emulsions; chloride/bromide emulsions have high light sensitivity, but the bromide/iodide emulsions have even higher sensitivity. Silver iodide is never used alone and used in a mixture with silver bromide, it normally constitutes 5% or less. Adding some silver iodide to fine-grained emulsions at low concentrations gives a higher sensitivity and contrast than pure silver bromide emulsions of the same grain size. Silver-halide crystals are cubical in shape and in each crystal a silver ion (Ag^+) is surrounded by six halide ions. The crystal normally possesses an excess of halide ions originating from the emulsion manufacturing process. Silver-halide grain sizes vary from about 10 nanometers for the ultra-fine-grained Lippmann emulsions to a few micrometers for high sensitive photographic emulsions (Table 1).

Table 1. Emulsion grain sizes

Type of emulsion	Average grain diameter [nm]
Ultra-fine-grained holographic emulsion	10-30
Fine-grained holographic emulsion	30-50
Fast holographic emulsion	50-100
Chlorobromide paper emulsion	200
Lithographic emulsion	200-350
Fine-grained photographic emulsion	350-700
Fast photographic emulsion	1000-2000
Fast medical X-ray emulsion	2500

Silver compounds are sensitive to light at various degree. Silver chloride is only sensitive to *violet* and *UV light.* Silver bromide absorbs light up to about 490 nm, and if silver iodide is added to silver bromide the sensitivity extends up to about 520 nm. Special *sensitisers* (dyes) must be added to the emulsion to make it sensitive to other parts of the spectrum. We say that a photographic material is *orthochromatic* if it is also sensitive to *green light.* If the material has been sensitised to the whole visible part of the spectrum, including *red light,* it is said to be *panchromatic*. It is also possible to make the material sensitive to infrared light (IR).

2.1.2 Holographic emulsions

The final quality of a holographic image will be the function of a number of factors, such as; the geometry and stability of the recording setup, the coherence of the laser light, the reference and object beam ratio, the type of hologram produced, the size of the object and its distance from the recording material, the recording material and the emulsion substrate used, the processing technique applied, as well as the reconstruction conditions. We know that, if during the reconstruction of the hologram the reference beam is identical with the recording reference beam, no image aberrations will occur. This applies also to circumstances when the reconstruction reference beam is a conjugate of the original reference beam (time-reversed). Theoretically, the holographic technique is the most perfect imaging technique in existence, since both the amplitude and the phase of the light wave scattered from the object are recorded. In practice, the holographic image is subject to certain limitations imposed by the recording material.

Three main factors will determine the resolution of a holographic image: the recording wavelength, the numerical aperture and the properties of the recording material itself. However, the following three points must be considered when discussing the attainable resolution of a holographic image:

- Ideally, the ultimate resolution should be independent of the properties of the recording material, and should depend only on the wavelength that was used for the recording as well as on the size of the recorded area of the material (the aperture) and the object distance.
- In practice, the limit on resolution may be set by the recording material, e.g., if it cannot record spatial frequencies above a certain limit.
- As regards aberrations introduced during the reconstruction of the hologram the following applies: If we assume that no aberrations are introduced by altering the reference beam, the only aberrations that will occur are then caused by the recording material itself.

A silver-halide emulsion must comply with certain requirements to be suitable for the recording of holograms. The most important of these concerns the resolving power of the material. The recording material must be able to resolve the highest spatial frequencies of the interference pattern created by the maximal angle θ between the reference and the object beams in the recording setup (Fig. 1).

If λ is the wavelength of the laser light used for the recording of a hologram, then the closest separation d_a between the fringes in the interference pattern (in air) is

$$d_a = \frac{\lambda}{2\sin(\theta/2)}$$

In the recording layer the fringe spacing d_e will depend on the refractive index n of the emulsion and is

$$d_e = \frac{\lambda}{2n\sin(\theta/2)}$$

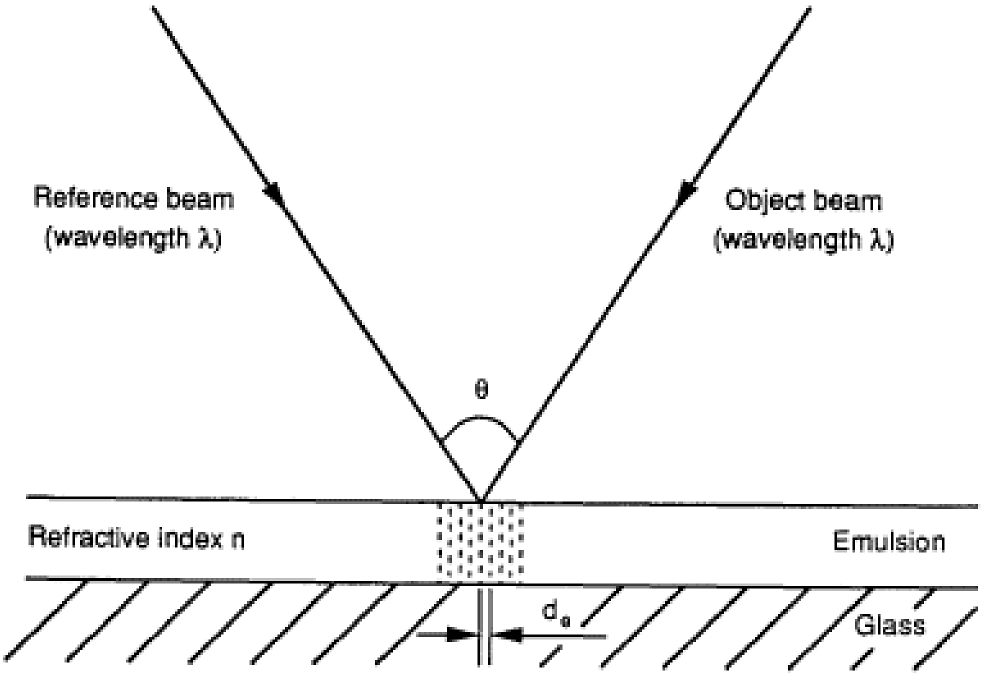

Fig. 1. Demand on resolution for recording a hologram. The recording material must resolve the highest spatial frequencies of the interference pattern created by the maximal angle C between the reference and the object beams in the recording setup.

One example of the resolving power needed in a practical situation using an emulsion with a refractive index of $n = 1.62$ is the following: A ruby laser with the wavelength $\lambda = 694$ nm and a recording geometry with the maximum angle $\theta = 25^o$ between the beams are used. This gives $d_e \approx 1$ μm, which corresponds to $v = l/d = 1000$ lines/mm; this is the minimum resolving power required. Close to its resolution limit the material will exhibit a low MTF and will thus make a low-quality hologram with poor fringe contrast and low signal-to-noise ratio. For high-quality holograms the resolution limit of the material must be much higher than the minimum value obtained according to the above formula. For a reflection hologram recorded in blue light ($\lambda = 400$ nm) with an angle of 180^o between the beams, a minimum resolving power of 7600 lines/mm is needed.

2.1.3 Image resolution

Theoretically, the resolution of the holographic image should be the true diffraction-limited resolution that can be obtained when the information is collected over an aperture equal to the size of the recording holographic plate. In principle, the larger the holographic plate the better the resolution will be. The limit is of the order of the wavelength λ when the dimension of the plate is infinite ($D \to \infty$). If the holographic plate is very large, however, the resolving power of the recording material will eventually limit the resolution of the image. If the resolving power of the recording material is sufficient, the diffraction-limited resolution can be obtained under the assumption that the high-resolution recording material is also perfect in that the position of the recorded interference fringes will not be changed during the processing of the material. In practice, a stable support for the emulsion (like a glass plate) will be needed and the processing methods applied must be such as not to affect the recorded fringe position in the emulsion, e.g., no fixing. The most limiting factor on the resolution of a holographic image is, however, in the form of distortions appearing in the emulsion. These aberrations are introduced by

- variations in the thickness of the recording medium before processing
- variations in the thickness of the recording medium produced during processing
- variation in the refractive index of the recording medium produced during processing
- deformations of the recording medium that will occur between recording and reconstruction.

2.1.4 Ultra-high-resolution emulsions of the Russian type

In Russia, the main interest has been in the production of reflection holograms of predominantly single-beam type, also called *Denisyuk holograms*. In this type of hologram, the grain size is of primary importance, which is why Russian holographers concentrate on emulsions containing very fine grains. In the emulsion which Denisyuk used for his first holograms, grain growth was slowed down by high concentration of KBr. The emulsion was made by Protas.[2] This emulsion had grain sizes of about 30 nm. TEA was used to sensitise the emulsion prior to use. The next step was to improve the old type of Lippmann emulsions. In this case it was possible to reduce grain sizes by instantaneous emulsification at a low temperature. Here, emulsification temperature was 32^0C and chilled alcohol was used. Rapid melting of the coagulated emulsion in a steam bath made reproducible results. A diffraction efficiency between 20 -24 % was obtained and the grain size was about 30 nm.

The experience gained enabled Protas to make the high quality plate LOI-2 (now called *PFG-02)*. Protas was able to slow down grain growth during emulsification by increasing the number of growth centers and introducing special growth inhibitors. The best Russian emulsion ever made is probably the one achieved by Kirillov *et al.*[3, 4] In their case, grain growth was hampered by the fact that in the emulsification process, a highly diluted solution was used and the emulsion concentration was increased by applying the method of gradual freezing and thawing. They used a rather diluted solution of emulsion containing 0.1 - 1 % gelatin and the following method. Just after the emulsion has been mixed, it is poured into a beaker and frozen at a temperature between -10^0C and -20^0C or even lower. The emulsion is kept in the beaker for 10 to 15 hours. A rapid freezing method is also suggested. Here, a thin layer of emulsion is poured into an already chilled tray. The frozen emulsion is then chopped into small noodles and put on a grid for thawing. To speed up this process, the frozen emulsion noodles are showered using cold water ($3\text{-}5^0$C). During this process, the emulsion is at the same time washed in order to remove unwanted salts. When the temperature of the emulsion increases (about $25\text{-}30^0$C) and becomes liquid, it undergoes a gentle ripening. The process of freezing and thawing can be repeated several times in order to increase the concentration of the emulsion. It has been verified that the silver content in the emulsion typically increases about ten times in this repeated process. The silver content of the emulsion is about 2 - 2.5 g/l at emulsification and after concentration 20 - 30 g/l. Sodium thiosulfate is added at a temperature of 30^0 to 32^0C for 5 to 10 minutes. Then, gold sensitizing takes place for 5 minutes as well as optical sensitizing. A 20% gelatin solution is added to the emulsion before it is coated on glass plates. This type of emulsion is used for the PE-2 plates (now called *PFG-03)* which has a grain size of about 10 nm.

2.1.5 Development of ultra-high-resolution emulsions

Russian type of emulsions can be processed in a very interesting way which will create very high-quality low-noise holograms. The most common procedure used for these types of holograms is based on rather diluted emulsions processed in semi-physical developers in such a way that silver particles of the order of 20 nm are obtained. A semi-physical developer contains not only a reducing agent but also a silver-halide solvent. In principle, during the development process Ag^+ ions are reduced to metallic silver in the latent-image centers. The number of these centers grows with increased exposure, whereas the number of silver ions in a given emulsion is constant, i.e. it is dependent solely on the concentration of silver in the emulsion and is independent of the exposure time. If the exposure time is short, few centers will be formed and silver ions will produce silver particles in the same way as when a conventional developer is used. The density of the emulsion will increase with exposure. However, above a certain exposure (the number of latent-image centers is high) due to the action of the semi-physical developer, the number of silver ions transferred to every center diminishes considerably, which results in silver particles of a smaller size than for conventional processing, and which in turn causes lower density of the emulsion, i.e. low absorption (colloidal silver). Some facts about such emulsions and their processing have been reported by Crespo *et al.*[5]

- The optimum silver concentration in the emulsion has been found to be 1.1 g/m^2 (emulsion thickness of about 10 µm).
- The development time in the Russian GP 2 developer is about 12 minutes *without agitation* at a temperature of 20^{0}C.
- The highest optical density can be obtained at the exposure of about 0.2 mJ/cm^2, while the optimal diffraction efficiency can be obtained at the exposure of about 0.6 mJ/cm^2 (3 times higher) when the optical density is reduced and colloidal silver is formed.
- The additional heat treatment also improved the diffraction efficiency, hardening the emulsion and thus making it less prone to shrinkage during processing.
- In general, the lower the concentration of silver ions in the emulsion, the longer the development time must be in order to obtain high diffraction efficiency.

From the discussion above it is clear why little success can be expected from the application of the semi-physical developing technique to the Western types of silver-halide materials (such as AGFA materials) which have a high silver content and rather large silver-halide grains. Normally, if this technique is applied to, e.g. AGFA materials, problems with silver precipitation on the emulsion surface combined with dispersion due to dichroic effects may occur.

It is also clear that due to the combination of very small grains in the emulsion and the demand for higher exposure in order to create colloidal silver during processing, Russian types of emulsions will be much less sensitive compared with commercial silver-halide emulsions produced by Western photographic companies. However, the advantage of using slower silver-halide emulsions is very high. The quality of the holograms recorded on such materials is higher than can be obtained on the commonly used AGFA materials of today. This fact should be encouraging to people who now have to learn how to use new and different silver-halide materials for holography. In the following a short survey of existing alternatives to AGFA materials will be given.

2.2 SLAVICH MATERIALS

In Russia the main producer of holographic materials is SLAVICH Joint Stock Co, Micron Branch, located outside Moscow.[6] The main differences between the SLAVICH and the KODAK and AGFA holographic materials are the grain size and the silver content in the emulsion. The Russian recording emulsions can have grain sizes as small as 10 nm and the silver content is usually one-half (about 0.25 g/cm^3) of the normal silver content in the Western materials. The glass plate sizes are: 4" by 5", 8" by 10", 288 mm by 406 mm, 609 mm by 812 mm. Glass substrate thickness between 1.8 mm and 5.0 mm. Sheet film: 200mm by 300mm, 102mm by 127mm, 203mm by 254mm. Slavich material can also be ordered as roll film. Later this year, SLAVICH intends to manufacture a roll film, 1 .2m by 10m.

Table 2. SLAVICH products

MATERIAL	Emulsion thickness [μm]	Spectral sensitivity [nm]	Sensitivity [mJ/cm^2] at 488	514	633	647	Resolving power [lp/mm]
Monochrome							
PFG-0l	10-12	633-647	-	-	0.1	0.1	3000
PFG-03M	7	633-647	-	-	3	3	10000
PFG-04 (DCG)	10-12	488-514	200	400	-	-	-
VRP (AH)		488-532	0.1	0.1	-	-	3000
VRP-M (No AH)	7	488-532	0.1	0.1	-	-	3000
Colour							
PFG-03C	7	480-647	1.5	1.2	1.0	1.0	10000

The PFG-01 plate is a potential replacement for pulsed ruby laser holograms. The PFG-03 is an excellent emulsion for reflection holograms recorded with red cw lasers.

2.2.1 Processing

The processing (development, fixing and bleaching) of Russian emulsions is slightly different from processing AGFA materials. The Russian emulsion is much softer than AGFA emulsions. A pre-hardening step is often necessary *before* development and bleaching can be performed. The following bath is used (6 minutes):

Distilled water	750 ml
Formaldehyde 37 % (Formalin)	10 ml (10.2g)
Potassium bromide	2 g
Sodium carbonate (anhydrous)	5 g
Add distilled water to make	1 l

Conventional developers, such as, e.g. *KODAK D-19*, can be used with SLAVICH products. For reflection work, the *CW-C2 Catechol developer*[7] works very well (after prehardening). Most rehalogenating holographic bleach baths work and, in particular, the *PBU-amidol bleach*[8] works very well, without any shrinkage of the processed emulsion (after prehardening). In addition, *PBQ* and *Ferric EDTA bleaches* work fine with these emulsions. Reversal bleaching using solvent bleaches seems to dissolve both exposed and unexposed silver halide grains, resulting in low diffraction efficiency and is not recommended for SLAVICH materials.

An interesting aspect of processing ultra-fine-grained emulsions is the possibility to use *colloidal development* and highly diluted developers. Colloidal silver grains formed in the emulsion during development are so small that very little absorption occurs (they act more like phase holograms). Holographers would probably like this method, since it produces holograms with high diffraction efficiency combined with very low scattering noise, applying a highly diluted (inexpensive) developer only. The Russian *GP-2 developer* (stock solution) is frequently employed for colloidal development:

Methylphenidone	0.2	g
Hydroquinone	5	g
Sodium sulfite (anhydrous)	100	g
Potassium hydroxide	5	g
Ammonium thiocyanate	12	g
Distilled water	1	1.

Working solution: 15 ml stock solution + 400 ml distilled water. Developing time at 20^0C is between 12 and 15 minutes without agitation.

After development the hologram can be fixed (which will introduce a shrinkage, and thus, a colour shift in a reflection hologram) or desensitized (with no shrinkage) in the following solution:

Phenosafranine ($C_{18}H_{15}CIN_4$)	300 mg
Methanol	0.5 1
Distilled water	0.5 1

Treatment time is about 3 minutes.

Very high-quality reflection holograms can be produced using the easy-to-use type of colloidal silver processing. For colour holography[9] the SLAVICH PFG-03c emulsion is the only commercial silver-halide emulsion that can produce high-quality large-format holograms.

2.2.2 Emulsion investigations

A few years ago, the SLAVICH monochrome materials were tested and compared with the AGFA products.[10] A single-beam recording setup for Denisyuk reflection holograms was arranged for recording a special test object. Helium-neon and argon ion lasers constituted the illumination sources. The processing of the silver-halide materials were performed according to the known techniques of obtaining high-quality amplitude and phase holograms.[1] For example, the three-step processing method of obtaining low-noise phase holograms introduced by Phillips was used for AGFA materials.[11]

The following silver-halide materials were investigated: AGFA Holotest 8E75HD, 8E56HD, lAE-reflex (another Russian emulsion), and SLAVICH PFG-03. Processing of the Western materials are indicated in the figure captions and the recipes are found in the references (e.g., Ref. 1). Phillips' three-step processing technique, fixation-free rehalogenating bleaching (PBQ, Fe-EDTA, and PBU-amidol bleaches), reversal bleaching (CW-C2 developer - dichromate bleach) were investigated. Russian materials were processed in the developer GP-2 and fixed or desensitised. The main results are summarized in Figs. 2 and 3.

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Scattering that occurs in the emulsion *during recording* limits both the resolution and the signal-to-noise ratio. The diffraction efficiency measurements of the recorded holograms are presented in Fig. 2. Here, the efficiencies are given as a function of the reference beam incidence angle. The shift of the Bragg angle towards smaller values clearly demonstrates the degree of emulsion shrinkage (in some cases expected as a result of fixing or reversal bleaching).

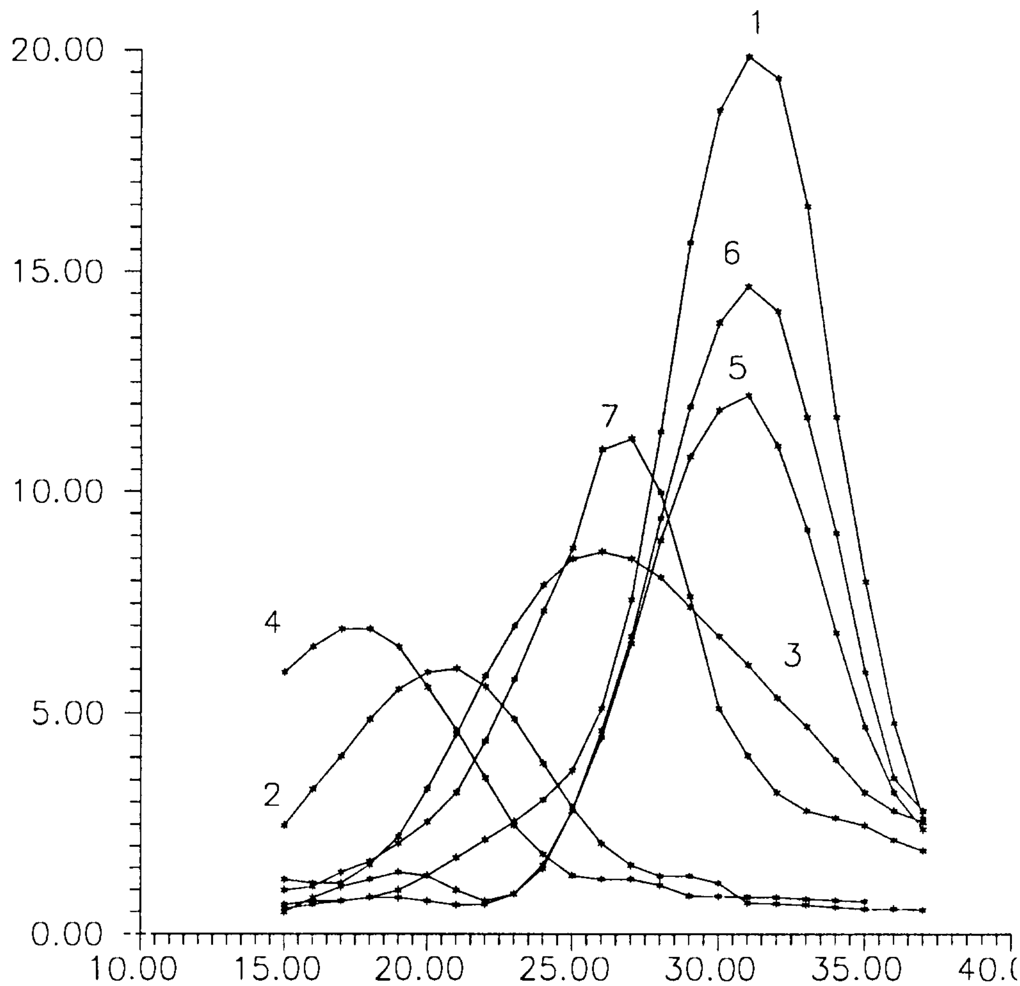
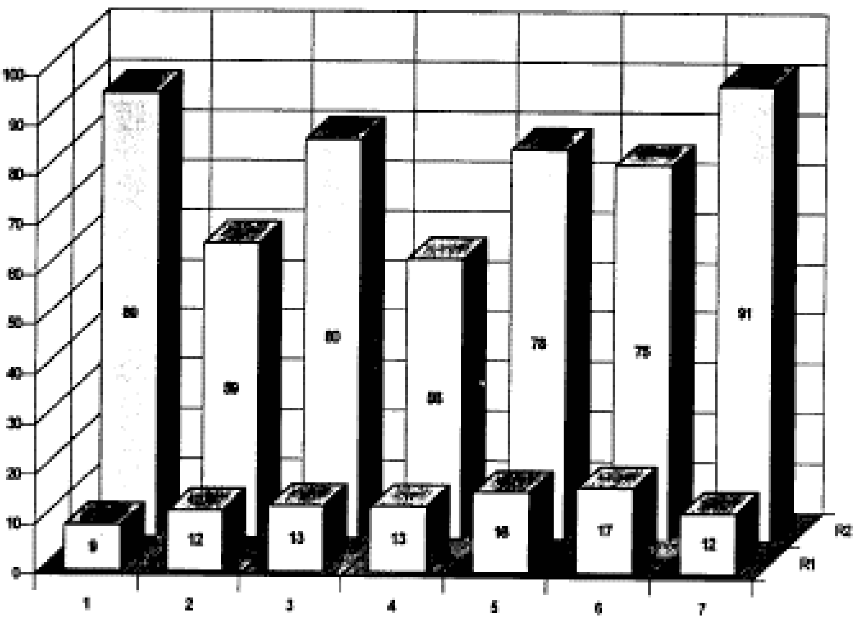


Fig. 2. The diffraction efficiency of the recorded test holograms as a function of the reference beam incidence angle.

1. Slavich PFG-03 processed in developer GP-2 and fixed.
2. Agfa 8E75HD processed in developer CW-C2 and bleached in a reversal dichromate bath.
3. Agfa 8E75HD processed in developer CW-C2 and bleached in a rehalogenating PBU-amidol bath and treatment in a sodium sulfite shrinkage bath for colour tuning.
4. Agfa 8E75HD processed in developer HOLODEV 602 and bleached in a reversal dichromate bath and Phillips' noise reduction redeveloping colloidal step.
5. Agfa 8E75HD processed in developer CW-C2 and bleached in a rehalogenating PBU-amidol bath and Phillips' noise reduction redeveloping colloidal step.
6. Agfa 8E75HD processed in developer CW-C2 and bleached in a rehalogenating PBU-amidol bath.
7. IAE-emulsion processed in developer GP-2 and fixed.

The noise evaluation was performed by contrast measurement in the black domain of the recorded image of the test object as well as of the object itself. Fig. 3. The Russian PFG-03 emulsion shows the highest diffraction efficiency as well as the lowest noise level. Among the holograms recorded on AGFA material, good results concerning both diffraction efficiency and signal-to-noise ratio, are obtained by the developer CW-C2 and the PBU-amidol bleach. In white-light reconstruction of the holograms of the test object the contrast was determined by measuring the intensity difference in the image between a perfectly absorbing black area and a perfectly white area both present in the test object. The contrast of the hologram image reconstruction was that presented in relation to the contrast of the same features measured at the object itself. This contrast at the object was set to be 100%. In relation to this value, the contrasts of the reconstructions for the same set of holograms described in Fig. 2 are presented at the back row R2 at Fig. 3. In the front row R1 the relative measurements of the scattering noise in the holographic reconstructions are presented. The figures appearing on vertical bars are percentage values of the maximum white that was detected in reconstruction. This light intensity, that is measured over the perfectly black areas of the reconstructed image of the object, is the measure of the scattering noise in the holographic reconstruction. It is observed that the highest contrast and lowest scattering noise are related to the Russian PFG-03 and lAE plates.

Fig. 3. Noise and contrast evaluation in holographic white-light reconstructions.



1. **Slavich** PFG-03 processed in developer GP-2 and fixed.

2. Agfa 8E75HD processed in developer CW-C2 and bleached in a reversal dichromate bath.

3. Agfa 8E75HD processed in developer CW-C2 and bleached in a rehalogenating PBU-amidol bath and treatment in a sodium sulfite shrinkage bath for colour tuning.

4. Agfa 8E75HD processed in developer HOLODEV 602 and bleached in a reversal dichromate bath and Phillips' noise reduction redeveloping colloidal step.

5. Agfa 8E75HD processed in developer CW-C2 and bleached in a rehalogenating PBU-amidol bath and Phillips' noise reduction redeveloping colloidal step.

6. Agfa 8E75HD processed in developer CW-C2 and bleached in a rehalogenating PBU-amidol bath.

7. IAE-emulsion processed in developer GP-2 and fixed.

2.3 HRT MATERIALS

In Germany, Richard Birenheide manufactures holographic emulsions in his HRT company.[12] The emulsion is harder than the SLAVICH products. At the present time only glass plates are manufactured. Later this year HRT will coat polyester film (100 µm or 180 µm thick). Initially, 500 or 600 mm wide rolls will be produced and cut to smaller sizes. The glass plate sizes are: 2.5” by 2.5”, 4” by 5”, 8” by 10”, 300 mm by 400 mm, 500 mm by 600 mm.

Table 4. HRT products

MATERIAL	Emulsion thickness [µm]	Spectral sensitivity [nm]	Sensitivity [mJ/cm^2] at				Resolving power [lp/mm]
			450	540	650	694	
Monochrome							
BB-700*	7	660-7 10	-	-	-	?	?
BB-640	7	580-650	-	0.3	-	-	5000
BB-520	7	480-540	-	0.2	-	-	5000
BB-450	7	410-470	0.2	0.2	-	-	5000
Colour							
BB-PAN	7	450/540/650	1.0	1.0	1 0		6000

* Proposed, not yet on the market

Recommended processing solutions for the HRT materials are an ascorbic acid developer followed by a rehalogenating copper sulfate bleach.

Developer:

Metol	4	g
Ascorbic acid	25	g
Sodium carbonate (anhydrous)	70	g
Sodium hydroxide	15	g
Distilled water	1	1.

Bleach:

Copper sulphate	35	g
Potassium bromide	100	g
Sodium hydrogen sulphate	5	g
Distilled water	1	1.

For reversal processing: a pyrogallol developer . dichromate bleach *(“pyrochrome”).*

More about silver-halide emulsions, hypersensitising, desensitising, and processing techniques can be found in Ref. 1.

3. Photopolymer Materials

An alternative to silver-halide material for holograms is the holographic photopolymers from E.I. du Pont de Nemours & Co. The monochrome materials (OmniDex®) are commercially available, but the panchromatic materials are still in the development phase. Photopolymer film is a very suitable recording material for mass replication by contact-copying holograms and HOEs from silver-halide masters. The photopolymer materials from DuPont are easy to use for holography. It has its special advantages of easy handling and dry processing (only UV-curing and baking). The emulsion thickness is 20 µm. After the exposure is finished, it has to be exposed to direct strong white or UV light for developing. DuPont recommends about 100 mJ/cm2 exposure at 350-380 nm. After that, the hologram is put in an oven at a temperature of 120^0C for two hours in order to increase the brightness of the image.

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