

Formation and characteristics of high resolution photographic plates

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High resolution photographic plates having a coating of exceedingly fine-grained photosensitive emulsions with sub-microscopic crystals of silver bromide in gelatin have been developed. The emulsions have been prepared by controlling various factors such as concentration of gelatin and reactants, emulsification time and temperature, mixing procedures and addition of inhibitor, hardener and sensitizer. Flatness uniformity, coating thickness, grain size, fog, contrast, sensitivity and resolution of the photographic plates have been determined.

High resolution photographic (HRP) plates have coating layers of ultrafine-grain photosensitive emulsions on its surface¹. These plates find applications in micro photography², in the reduction of charts, masters, documents, photographs or precision scales to minute or even to microscopic scale³, in the making of exact scales, graticules, scales of diffraction grating type and interference filters⁴, in the recording of nucleo-physical phenomena⁵, in astrophotography⁶ and spectrophotography⁷, in the making of masters for the production of microelectronic integrated circuits⁸, in holography⁹, etc.

The photosensitive layer on the HRP plate not only has to have a fine structure, it must also be free from spreading of the record of an image thrown upon it. This is probably the reason why the silver halide emulsions still hold the field in these applications. Other photosensitive layers cannot match the photographic performance of silver halides. The extremely low solubilities of silver halides render them for all practical purposes fixed in position. In spite of wide research¹⁰ no compound or substance has yet been discovered which is anything like as sensitive to light as bromide of silver, yet extreme sensitiveness has so far been obtained only when it is subjected to the influence of gelatin.

The early photographic emulsions¹¹ had a more or less fine grain and the finest grain plates were, however, those which could produce "interference colours" by recording standing waves of light. Gabriel Lippmann produced the first permanent colour photographs by the method of interference colours, which involved the incidental preparation of finest-

grained silver chloride-collodion plates¹². In recent years the fine-grain properties of Lippmann emulsions have been made available in the form of high resolution photographic plates for a wide range of applications¹³.

It may be mentioned that it is almost impossible to prepare a Lippmann emulsion and remelt at a later date for coating because of grain growth leading to loss of the most essential property of the emulsion. Hence the experimenter with the Lippmann process is forced to prepare his own emulsions. In spite of various commercial imported HRP plates now available, it may be useful to coat one's own plates in order to obtain a finer grain or another thickness.

The manufacture of photosensitive photographic films/plates/papers is a monopolised industry, the technology of their production being a closely guarded secret. Due to lack of published information, the actual technique of making of ultrafine-grain photosensitive emulsions is not known. There is no firm in India which manufactures high resolution photographic plates. In the present investigations, attempts have been made to find out technical details for the preparation of ultrafine-grain photosensitive emulsions and to determine the photographic characteristics of HRP plates made from these emulsions.

Experimental Procedure

The steps involved in the preparation of ultra-fine grain high resolution photographic plates are listed in Table 1.



Fig. 1—Electron microscope enlargement ($\times 100,000$) of the silver bromide grains of emulsion type 'C'

Emulsion Preparation

Making of emulsion is the most critical stage in the preparation of HRP plates which ultimately controls the photographic characteristics of the plates. The eight main components of emulsion making recipe are: photographic quality gelatin (George Nelson Dale & Co. Ltd., England), silver nitrate (GR, ACS, ISO, E. Merck (India) Ltd.), potassium bromide (GR, ACS, E. Merck (India) Ltd.), 6-nitrobenzimidazole (inhibitor; Koch-Light, England), chromium(III) potassium sulphate dodecahydrate (chrome alum) (hardener; GR, ACS, E. Merck (India) Ltd.), 3-3' diethyl 4-5, 4'-5'-dibenzothiacarbocyanine iodide (sensitizer, Koch-Light, England), methanol (GR, ACS, ISO, E. Merck (India) Ltd.) and double distilled water, all of high purity.

The main reactants of emulsification process are potassium bromide and silver nitrate. During emulsification, precipitation of finely divided solid phase of silver bromide takes place by a double decomposition reaction between silver nitrate and potassium bromide in presence of protective colloid gelatin. The basic photosensitive substance of the finished product is silver bromide.

Photographic quality gelatin of 125 bloom strength in granulated form is used. It must be free from substances which might cause fogging in the finished emulsions. The requisite amount of dry gelatin is dissolved by soaking in desired volume of cold double distilled water kept in a beaker. The beaker is placed in cold water bath and the heater and stirrers turned on, the thermostat being set at appropriate temperatures. Complete solution of the gelatin takes

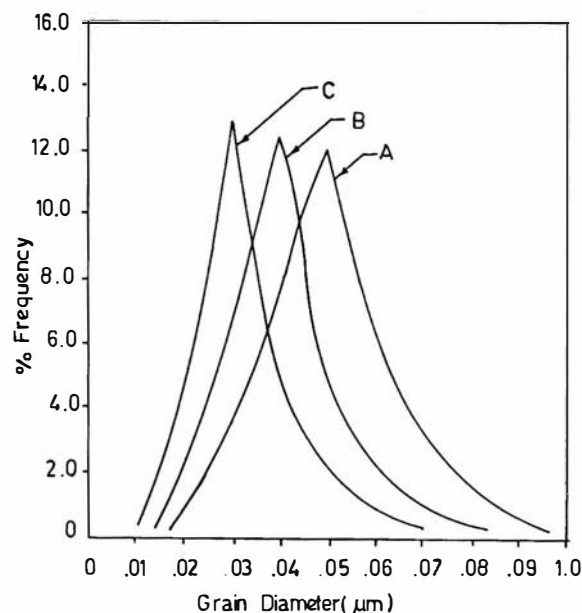


Fig. 2—Size distribution curves of diameters of the grains of photosensitive emulsions

at least an hour, it is desirable to allow an hour and half.

Gelatin solution is filtered through fine muslin cloth. Two thicknesses of good quality fine muslin cloth which has been boiled in order to remove any sizing material, is used. Filtration is done to remove small fragments of insoluble matter, string fibre, dust and dirt if any present in gelatin. Gelatin solution is next poured into a clean beaker which can hold two to three times as much liquid. The beaker is next placed in a thermostatically controlled water bath at desired temperatures.

Formation data of three typical photosensitive emulsions 'A', 'B' and 'C' are listed in Tables 2 and 3. Concentrations of gelatin, silver nitrate, potassium bromide, inhibitor, hardener and sensitizer employed in the making of photosensitive emulsions are shown in Table 2. Table 3 depicts the emulsification temperature, advance of silver nitrate and emulsification rate during emulsification process, gelatin/silver ratio and silver bromide content of photosensitive emulsions.

During emulsification, for emulsion type 'A', 'B' and 'C', the temperatures of gelatin solutions are kept as 33°C, 30°C and 27°C respectively and those of silver nitrate and potassium bromide as 21°C. A temperature gradient is kept between solutions of gelatin and main reactants in order to achieve finer-grain size during emulsion making.

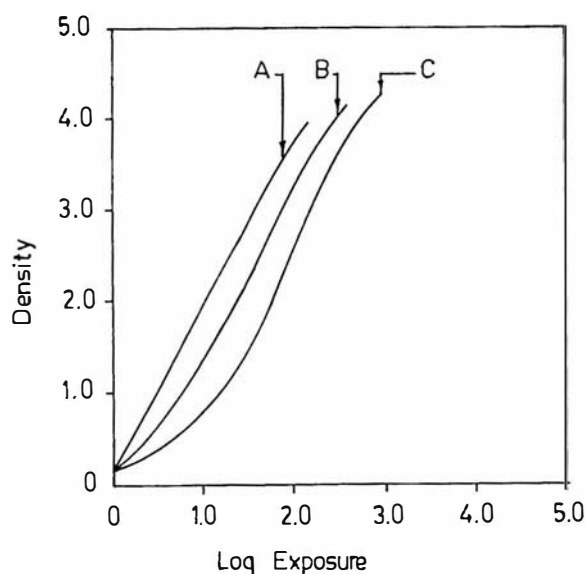


Fig. 3—Hurter and Driffield characteristic curves of photosensitive emulsions

A novel double-jet technique is employed for mixing of silver nitrate, potassium bromide and gelatin solutions. Aqueous solutions of potassium bromide and silver nitrate are poured drop by drop, at the rate of 2 drops per second from burettes to the gelatin solution kept in a beaker of 250 mL at appropriate temperatures. The beaker is placed in a magnetic heating bath (Remi, India). The gelatin solution is stirred all the time with an AC direct drive laboratory stirrer (Remi, India), as vigorously as possible without producing froth or bubbles. The maximum agitation speed is 1100 RPM. The rate of stirring effects the initial grain size, at higher stirring rates aggregation of silver bromide particles is prevented and the particle size distribution is more uniform.

The resulting suspension of silver bromide in gelatin appeared nearly clear to milky depending on its concentration, but a thin layer of emulsion taken out on a glass rod always appeared nearly clear with a bluish opalescence which is a rough indication of fineness of grains.

In this unique technique of emulsification, reactants are at a high and approximately constant dilution throughout the whole course of precipitation, since they are continually being removed by their own reaction as fast as they are added. The accumulation of the soluble product of reaction, potassium nitrate, fortunately appears to have no effect on the kinetics of reaction.

Table 1—Flow chart of making of ultrafine-grain high resolution photographic plates

* Emulsion preparation
- Making of solutions (Gelatin, silver nitrate, potassium bromide, inhibitor, hardener, sensitizer)
- Emulsification (Temperature, time, mixing procedure, vigorous stirring)
- Addition of finals (Inhibitor, hardener, sensitizer)
- Filtration
* Coating
- Substratum layer, dip-coating
- Emulsion layer, flow-coating
* Chilling
- Setting of emulsion
* Washing
- Removal of by-products
* Drying
* Testing (Flatness uniformity, coating thickness, grain size, fog, contrast, sensitivity, resolution)

So long as the addition of the reactants is slow, experiments have shown that concentrations of both gelatin and reactants may be varied between very wide limits with hardly any effect on the grain size of the precipitate. It has been observed that small advance of silver nitrate solution over potassium bromide solution, e.g. 0.3 to 1.0 mL of appropriate concentrations, resulted in the production of a finer grain as was evaluated simply by looking through the emulsion poured into a test tube and estimating the transparency. Measurements of the particle size of the grains by means of electron microscope enlargements confirmed this result. It may be related to the solubility of silver bromide at varying temperatures and concentrations of silver.

The emulsification process and all subsequent parts of the method must be carried out in safe lights of suitable colour so that the photosensitive emulsions are not fogged. A deep orange red safe light is generally suitable for orthochromatic emulsions. However, for panchromatic emulsions, the minimum amount of green light is best, not because it is "safe", but because it is the most efficient visually and consequently one can manage with the minimum amount of radiant energy.

Table 2—Formation data of ultrafine-grain silver bromide emulsions

	Emulsion 'A'	Emulsion 'B'	Emulsion 'C'
Gelatin	1.6 g in 60 mL water	1.4 g in 50 mL water	2.0 g in 80 mL water
Silver nitrate	1.0 g in 10 mL water	1.0 g in 10 mL water	3.5 mL of 1.0 g per 10 mL aqueous solution
Potassium bromide	0.75 g in 10 mL water	0.75 g in 10 mL water	3.5 mL of 0.76 g per 10 mL aqueous solution
Inhibitor	0.05 g in 1 mL methanol and 1 mL water	0.05 g in 1 mL methanol and 1 mL water	0.0175 g in 1 mL methanol and 1 mL water
Hardener	0.8 mL of 1% aqueous solution	0.7 mL of 1% aqueous solution	1 mL of 1% aqueous solution
Sensitizer	1.5 mL of 0.5% methanol solution	1.5 mL of 0.5% methanol solution	1.3 mL of 0.2% methanol solution

The main operation of precipitating silver bromide having been carried out, there are three more operations in nature of small additions of finals which are, however, essential to the practical effectiveness of the final product. The first is addition of solution of 6-nitrobenzimidazole (inhibitor) in methanol and water. The concentration employed is shown in Table 2. Chemical adsorption of inhibitor on silver bromide forming stable complex compounds with silver bromide and oriented adsorption layer of the compounds suppress grain growth and prevent the formation of fog since these complex compounds are not readily decomposed or reduced to silver.

The second addition of finals is aqueous solution of chrome alum (hardener) to the emulsion (Table 2). Addition of hardener in the emulsion results in cross-linking of neighbouring gelatin macromolecules by their active groups (amino, hydroxyl, etc.), with different degrees of tanning of the final emulsion layer. Hardening prevents frilling and even detachment of emulsion layer during processing after exposure.

The third addition is a colour sensitizer. Plain silver bromide is not appreciably sensitive to wavelengths longer than those corresponding to the blue or violet end of the spectrum and it is required to extend and enhance the sensitivity of the silver bromide grains towards the desired end of the spectral regions. This is done by adding certain organic dyes to the emulsion. Sensitizing dyes are adsorbed on the silver bromide grains and sensitize the emulsion in the spectral

regions where they absorb light energy. This light energy absorbed by the sensitizer is transmitted by an electronic-ionic mechanism to the emulsion grains¹⁴. As a result, when the emulsion layer is exposed latent image centres may be formed on their surfaces by the action of the long-wave length region of the spectrum, to which they are themselves insensitive.

It is apparent that for full sensitization each silver bromide grain is completely covered with a single layer of adsorbed dye molecules, no more, no less. This means that the proportion of dye to silver bromide depends on the state of division of the latter and it has been found by experiments¹⁵ that ultrafine-grain emulsions need some 20 to 30 times as sensitizing dye per unit quantity of silver bromide as do ordinary emulsions. This leads to a practical difficulty with some sensitizers, that they cannot be dissolved in sufficient concentrations in methanol (the usual solvent) to sensitize fully an emulsion which contains a high proportion of silver bromide. This difficulty has not yet been fully resolved, fortunately it does not, in practice, lead to any important loss of efficiency.

The appropriate amount of 3, 3'-diethyl, 4, 5, 4', 5'-dibenzothiacarbocyanine iodide (sensitizing dye), dissolved in methanol (Table 2) is added to the emulsion after the addition of inhibitor and hardener and immediately before the emulsion is finally filtered and coated.

Emulsion thus made is filtered through a previously boiled fine muslin cloth in order to make

HRP plates of utmost cleanliness. Filtration assists in the removal of dust and dirt (in chemicals) and in breaking up of clumps or aggregates of silver bromide grains, which are always formed to a more or less extent in the process of making of emulsion.

Coating

To avoid grain growth of silver bromide due to ripening, the minimum interval should elapse between making the emulsion and coating and chilling.

Before coating of emulsion on glass plates, a substratum coat of 1.0% gelatin and chrome alum (0.5% of dry weight of gelatin) solution is applied on the glass plates by dip-coating technique. Dip-coating is done by dipping the chemically cleaned glass plates into a dish containing the substratum solution of gelatin and chrome alum and wiping the other sides of the plates with a muslin cloth where the emulsion coating is not to be done. After substratuming, the glass plates are dried by racking them at an angle in a nearly vertical position in a perfectly dust-free cupboard.

The time-honoured method of coating photographic emulsion on glass plates is by flow-coating technique¹⁶. Flow-coating means pouring across a plate. Glass plates are levelled as accurately as possible so that the emulsion can attain more or less of uniformity in thickness before setting. A setting slab of metal with top machined flat and through the body of which ice cooled water is circulated is employed. Substratum coated glass plates are slightly warmed somewhat (so that the warm emulsion may be flowed on evenly) and placed on accurately levelled chilled metallic setting slab. In order to obtain desired coating thickness of emulsion on drying, necessary amount of warm emulsion is poured on the warmed substratum coated glass plate, it is flowed on and spread with a glass rod so that it attains a uniform thickness before cooling to setting point by loss of heat to the setting chilled slab.

Chilling

Emulsion coated glass plates are allowed to cool and set by gelation on the accurately levelled chilled setting slab. The emulsion layer sets to a firm jelly by gelation within 30 to 60 seconds.

Washing

Next step is washing i.e. removal of by-products. The soluble product of the precipitation reaction, potassium nitrate and excess of potassium bromide

Table 3—Formation data of ultrafine-grain silver bromide emulsions

	Emulsion 'A'	Emulsion 'B'	Emulsion 'C'
Emulsification temperature	Gelatin, 33°C AgNO ₃ 21°C KBr	Gelatin, 30°C AgNO ₃ 21°C KBr	Gelatin, 27°C AgNO ₃ 21°C KBr
Advance of silver nitrate (mL)	0.3	0.5	1.0
Emulsification rate	2 drops per second	2 drops per second	2 drops per second
Gelatin/silver ratio	1.6	1.4	5.7
Silver bromide (g per mL)	0.013	0.015	0.004

must be washed out of the coated plate before they are allowed to dry appreciably; otherwise they may crystallize out and spoil the smoothness of the surface of the emulsion layer and would destroy the photographic characteristics of the emulsion. Chilled coated glass plates are washed with cold deionized water, temperature not exceeding 7°C, to remove the by-products from the emulsion. To preserve the cleanliness of the plates, the coated glass plates are finally washed with cold double distilled water.

Drying

Emulsion coated washed glass plates are air-dried in a very gentle stream of dry air in dust-free dark cup-boards so that the gelatin of the emulsion layer dries down with the minimum of strain and distortion.

The photographic plates having being finally dried may be stored for long periods of time without deterioration, especially if packed in pairs with the emulsion layers face to face.

Considerable care is necessary to maintain cleanliness in the making of HRP plates and it is essential to carry out all the operations, right from emulsion preparation to drying stages, in temperature and humidity controlled dust free atmosphere.

The HRP plates having been made, the plates must be placed at once in clean containers from which they are only removed in another dust-free temperature and humidity controlled space for testing.

Results and Discussion

The main characteristics of ultrafine-grain high resolution photographic plates are the flatness

Table 4---Photographic characteristics of high resolution photographic plates

	Emulsion 'A'	Emulsion 'B'	Emulsion 'C'	Agfa-Gevaert Millimask Plate
Flatness uniformity	>70%	>70%	>70%	> 70%
Coating thickness (average)	6 μm	6 μm	6 μm	6 μm
Mean diameter of grains	0.05 μm	0.04 μm	0.03 μm	0.06 μm
Fog density	< 0.1	< 0.1	< 0.1	< 0.1
Contrast	1.8	1.9	2.0	High
Sensitivity	0.67 ASA	0.55 ASA	0.42 ASA	Orthochromatic
Resolution	>3000 l/mm	>3000 l/mm	>3000 l/mm	~3000 l/mm

uniformity, coating thickness, grain size, fog, contrast, sensitivity and resolution. The data of these properties of HRP plates made from photosensitive emulsions 'A', 'B' and 'C' and of Agfa-Gevaert Millimask Plates are shown in Table 4.

Flatness uniformity in percent may be defined as the ratio of minimum coating thickness and maximum coating thickness multiplied by 100¹⁷. Flatness uniformity of emulsion layer of HRP plates have been found to be greater than 70 percent (Table 4).

Emulsion layer coating thickness is measured by Talysurf-4 Instrument (Rank Taylor Hobson, Leicester, England) which uses a stylus to trace surface irregularities to 0.03 μm in depth. The depth of a scratch that reaches the glass substrate is measured by this instrument. Average emulsion layer coating thickness of HRP plates have been found to be 6 μm (Table 4).

Grain size of the silver bromide emulsions are determined by means of scanning transmission electron microscope (Amre, USA). A typical electron microscope enlargement (X 100, 000) of the silver bromide grains of emulsion type 'C' has been shown in Fig. 1. Electron microscope enlargements of silver bromide crystals of the coated emulsion layers of emulsions 'A', 'B', and 'C' and measurements of the dimensions of 1000 grains give the following mean values with their standard deviations:

- Emulsion 'A' : $\bar{d} = (0.0498 \pm 0.0095) \mu\text{m}$
- Emulsion 'B' : $\bar{d} = (0.0401 \pm 0.0082) \mu\text{m}$
- Emulsion 'C' : $\bar{d} = (0.0308 \pm 0.0071) \mu\text{m}$

Size distribution curves of the diameters of the grains of emulsions have been shown in Fig. 2, where percent of frequency has been plotted against grain diameter. It has been observed that emulsion 'A' has mean diameter of grains as 0.05 μm; emulsion 'B' as

0.04 μm and emulsion 'C' as 0.03 μm. It may be noted that the particle size distribution is nearly Gaussian.

Hurter and Driffield (H&D) characteristic curves of the emulsions 'A', 'B' and 'C' have been shown in Fig. 3. Here optical density has been plotted against logarithm of exposure. Exposure to emulsion layer is given by Kodak Sensitometer Type 6 (France) using a light source and an optical step wedge with 21 steps having densities ranging from 0.15 to 3.0, the step increment being 0.15. This wedge tablet provides with an intensity scale of exposure. For development of coated emulsion layer, a standard developer (Kodak D-72) with constant temperature (27°C) is used. The time of development (4 min) is also controlled. Optical density (logarithm of opacity) is measured with the help of densitometer (Beckman, USA). The exposure is determined by the Eq.(1)¹⁸

$$\log_{10} E_s = \log_{10} E_o - D_s \qquad \dots (1)$$

where E_s is the illuminance or intensity of light source of sensitometer in lux, E_o is the illuminance or intensity of light source in lux without step wedge and D_s is the density of each step of sensitometric tablet.

From H&D characteristic curves fog, contrast and sensitivity (speed) of the emulsions have been determined. The unexposed areas on HRP plates on development may possess some density (opacity). This is known as fog¹⁹. The unexposed silver bromide grains on development may be reduced to finally divided metallic silver jet black in colour. The reduced silver is known as fog silver. Some of the density may be due to the plate base or the gelatin layer coated upon the base. Some density may result from development of unexposed silver bromide grains (fog) or from the reduction of indiscriminantly exposed silver bromide (from lens flare). The density never reaches zero because the base glass plate

absorbs some light and also because some silver bromide grains are always developable due to sensitization by heat and or background radiation. These developed background grains are called fog. Minimum density of HRP plates is the sum of fog and base density. Base density may be due to the glass plate base and gelatin layer on it. It has been observed that the fog densities of three photosensitive emulsions 'A', 'B' and 'C' are less than 0.1 (Table 4).

Contrast may be defined as the tonal range between the lightest and darkest areas of a photographic image or print²⁰. Contrast is the slope of the straight line that joins specified minimum and maximum densities on the H&D characteristic curve of the HRP plates. It is therefore an adaptation of average gradient, assuming a specified "average" image luminance range. Minimum density point lies on the arc of a circle having a radius of 0.2 density units above base plus fog density. The maximum density point lies on the arc of a concentric circle that has a radius 2.0 greater than the smaller circle. Contrast of photosensitive emulsions 'A', 'B' and 'C' have been found to be 1.8, 1.9 and 2.0 respectively.

Sensitivity or speed gives a measure of minimum exposure that gives a satisfactory print on a photographic material so that images could be recorded at low light levels²¹. Arithmetic speed in ASA is equal to $0.8/E_m$ where E_m is the exposure in lux seconds that is required to produce a density of 0.1 units above fog plus base density corresponding to a point "m" on the H&D curve. In the formula 0.8 is taken because H&D curve is considered to be standard when the change in density between 0.1 above base plus fog and 0.9 above base plus fog occurs when the logarithm of exposure changes by 1.3 units. The ASA speed of photosensitive emulsions 'A', 'B' and 'C' have been found to be 0.67, 0.55 and 0.42 respectively (Table 4).

Resolution is the ability of a photographic material to record fine details sharply and distinguishably or may be defined as the maximum number of parallel and equidistant lines and spaces per millimeter which can be distinctly recorded on the photographic material²². Finer the grain size of the silver bromide in photosensitive emulsions, higher the resolution. The resolving power is defined as the reciprocal of spacing (line plus space called line pair) and is usually quoted as lines pair per mm or more commonly lines per mm.

The resolution of HRP plates has been determined with the help of scanning transmission electron microscope using holographic techniques. Resolution ($1/d$) is determined by the Bragg's equation²³ indicated by Eq. (2),

$$d (\sin \alpha + \sin \beta) = n \lambda \quad \dots (2)$$

where d is the spacing of interference fringes, α is the angle of incidence, β is the angle of diffraction, n is the order of diffraction and λ is the wavelength of light. The resolution of photosensitive emulsions 'A', 'B' and 'C' has been found to be greater than 3000 lppm (Table 4).

It has been observed that the photographic properties of indigenous HRP plates have been found to be quite comparable to those of imported HRP plates obtained from leading world manufacturers like Kodak, Agfa-Gevaert and others (Table 4).

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