Micrography — The Making of Microscopic-size Photographs

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Summary
The term Microphotography relates to the manufacture of minute photographs of conventional-size objects or documents. Microphotographs are more popularly known as 'Microdots' and this term gives some idea of their size. The term Microphotography must not be confused with the term Photomicrography which is the production of conventional photographic prints or transparencies of very small items normally observed under the microscope. The size of a microphotograph may be such that the whole page of a newspaper can be reduced to the size of a dot on a piece of paper and yet can be easily read under a microscope. The smallest scale microphotographs will require the best of objectives and lighting to resolve and in practice a scale of a few millimeters would be used for a whole newspaper page.

Manufacturing methods

THE MICROPHOTO is produced with ultra-fine grain emulsion coated on a 3″ × 1″ glass slide using the apparatus to be described (shown at the 1985 Quekett Exhibition). The image is reproduced on a 35 mm or other format film, as a negative and this is used to produce the final microscopic positive. The negative is produced as in a conventional camera, but a document copying quality, fine grain film is essential. Once available the negative is mounted and illuminated in the copying apparatus and the exposure made on the ultra-fine grain coated slide.

The ultra-fine grain emulsion can be purchased from Kodak as 'ready coated' plates but these are expensive and only available in larger sizes. Only a minute portion of the emulsion is required, and the slides have to be cut and edged in a dark room. The alternative to this expensive option is to manufacture one's own emulsion and coat the central area of conventional microscope slides. This is the technique to be described below.

The photographic emulsion

The method to be described is the result of several years development based on details provided by Crawford (1954). Reference to this paper was obtained from the book on Micrography by Stevens (1942) which is recommended reading before embarking on the manufacture of microphotographs. The method is not claimed to be the ultimate, but it is a method that can be used in the home with relatively simple home-made equipment and basic dark-room facilities and skills. It is capable of producing grain sizes lower than those obtainable from commercially available emulsions.

Chemicals required

With one exception the chemicals required are easily obtainable and inexpensive, as only very small quantities are required.

1. Gelatine, cooking quality proved to be exceptionally pure. Diluted 4 g in 80 ml of distilled water.
2. Potassium Bromide diluted to 1.5 g in 10 ml of distilled water.
3. Silver Nitrate diluted to 2 g in 10 ml of distilled water.
4. Chrome Alum diluted to 2 g in 100 ml.
5. Sensitizer*—0.5 g in 100 ml consisting of 20 ml of distilled water and 80 ml of alcohol.

All the solutions must be filtered using 0.5 μ 'Millipore' filters which leave virtually no residue. These filters are small discs about 10 mm diameter which can be mounted in a holder on the end of a plastic syringe and the liquid squeezed through. The solutions must be stored in bottles thoroughly washed in distilled water. One problem is that the gelatine solution must be filtered hot and a coarser filter paper is necessary to facilitate its passage through the filter. All fine particles must be removed as they will show up on the final image.

All the solutions, with the exception of the gelatine, can be stored for long periods in sealed containers, preferably in the dark and even the gelatine solution can be stored for several months if 10% of the liquid is made up with alcohol after filtering.

Mixing the Emulsion

The main difference between ultra-fine grain emulsions and conventional emulsions is in the mixing process and the coating of the slide. The sensitive grain particles 'grow' as the chemicals are mixed and the secret is to prevent the particle from joining one another and thus giving larger particles. This is achieved by rapidly stirring the gelatine base as the chemicals are added to it. Indeed, the more rapidly it can be stirred the finer the grain size although the slower will be the speed of the emulsion. These ultra-fine grain emulsions are very slow indeed, even compared with photographic bromide papers.

The basic emulsion is formed by adding the potassium bromide and the silver nitrate to the gelatine. The chrome alum is used as a hardener to ensure that once set, the emulsion will not redesolve when washed, developed or fixed. This can be added immediately after the emulsion is formed together with the sensitizer. As well as mechanically stirring the emulsion it must be maintained at a constant temperature on a hot-plate or water bath to ensure that the gelatine does not set. Also the two chemicals must be added to the gelatine exactly one drop of each at equal times until completely used.

This process together with later ones involves the construction or adaption of various pieces of simple apparatus to achieve the required results. The mixing and temperature control apparatus consists of a thermostatically controlled hot-plate on which stands a small water bath holding the gelatine container, thus ensuring even heating of the gelatine and the resultant emulsion. A mixing rod with a small 'T' end, manufactured from a plastic knitting needle and mounted on the end of a constant speed motor, is used for stirring.

*The sensitizer is available under the Eastman Kodak trade name 'Pinaflavol' and was obtained in 5 g quantity several years ago via Kodak at Hemel Hempstead. The chemical name on the container was 2-(p-Dimethylaminostyryl)-1-ethylpyridinium iodide.

Any small motor can be used but a constant speed (e.g., synchronous clock motor at 200 r.p.m.) version ensures consistent results. Two 'droppers' each containing 0.5 ml of the two chemicals are mounted on spring 'Terry' clips for easy removal and with their ends pointing into the gelatine container either side of the central stirring rod. 4 ml of gelatine is added to the container and the dimensions should be such that the level is as deep as possible and the stirrer reaching to the sides to ensure even mixing.

With the gelatine up to temperature (about 35°C to 40°C) the stirrer is switched on and after a minute the 0.5 ml quantities are added, a pair of drops at a time until all of the solutions are used. A little practice before hand is needed and after a while it becomes easy to achieve exact matching of the drops.

Whilst still stirring, six drops of the hardening solution should be added and five drops of the sensitizer. After a few seconds the stirrer can be switched off and the solution is ready for coating. Although all the chemicals can be prepared under normal illumination conditions, the mixing and all further operations on the emulsion must be carried out in a darkroom or equivalent using only the illumination from a normal deep red photographic darkroom lamp.

Coating the slide with Emulsion

In coating and preparing the slide for exposure several other items of equipment need to be acquired or made. In order to justify the time spent on making slides it is better to prepare a quantity at one session. In order to do this a couple of holders are required that will contain about 25, 3" × 1" slides each to hold the slides during the washing and drying processes. An added advantage would be some form of light-tight box into which a small fan can be fitted to speed the drying of the coated slides.

Once the emulsion had been prepared it must be coated on the slides immediately otherwise grain clumping will gradually occur and render the emulsion useless. Only about 15 to 30 minutes is available before the emulsion goes milky, indicating a coarse grain structure. When first prepared, the emulsion is almost clear, as are the finally coated slides. The temperature of the hot plate should be turned down to 30°C after the emulsion is finished and allowed to stand on the hot-plate while the slides are coated. The lower temperature slows further grain growth whilst still being high enough to prevent the gelatine from setting.

As the final image is so tiny (for example the letters in a document may be only tens of microns high), the final thickness of the emulsion should only be a fraction of this, *i.e.*, perhaps 5 μm. The emulsion is applied using a glass dropper rod about 3 mm diameter. The slide is centred on a ringing table and rotated fairly slowly. The end of the rod is dipped in the emulsion, applied to the centre of the slide and drawn out slowly as the slide rotates until the emulsion at the edge almost disappears.

The slide is then immediately transferred to a cold plate, either glass or metal, cooled in a refrigerator (*e.g.*, 10°C), which sets the emulsion. After a few minutes the slides can be transferred to the storage frame mentioned previously.

One other consideration is that the slides must be scrupulously clean and free from dust. One method which has been attempted is to coat the cleaned slides with a strip of Sellotape which is peeled off just prior to coating, thus removing all dust particles with it. The storage frames containing the coated slides, which should be held horizontally, are transferred to the refrigerator (not the freezer compartment) for a further 10 minutes to ensure that the emulsion has set. The slides are then placed in a bowl of cold water, slowly agitating them to wash the emulsion. Note that all the above steps must be carried out in the dark or using a red safe light, taking care that the refrigerator does not have an internal light. The final gentle wash should be carried out in distilled water and the frames and slides transferred to the drying container.

Construction of the drying chamber
The drying container was made from a discarded biscuit tin with light baffles soldered inside, using further pieces of tin plate. This permits a stream of air to pass through the container without allowing light in. This allows the drying process, which may take several hours, to be carried out in normal lighting conditions. A small D.C. motor fitted in a tube on the lid of the container and driving a fan provides a gentle flow of air through the container.

After drying, the slides can be transferred to a light-tight storage box ready for use. No experiments have been carried out on the life of the coated slides as they tend to be used up within a week of manufacture.

The Copying Apparatus
The copying apparatus can consist simply of an adapted microscope or a specialised piece of equipment manufactured for the purpose. The equipment described below is in the second category. In photomicrography the slide to be photographed is placed on the microscope stage and suitably illuminated. Using the microscope, the image of the slide is projected on to the film plane of the camera mounted above the eyepiece position. Either the objective only, or the objective eyepiece and camera lens is used to form this image. Microphotography is the reverse of this procedure in that the negative to be copied is mounted above the eyepiece position and illuminated with strong, even illumination. Using conventional microscope optics, a tiny image is formed on the stage of the microscope where the ultra-fine grain photographic emulsion is placed. This is, of course, exactly the reverse procedure of photomicrography.

The exposure is made simply by switching the illumination on and off, exposure times being tens of seconds due to the slow emulsion speed. The greatest problem encountered is the need to ensure that the image is in focus in the plane of the emulsion. Various mechanical methods have been devised that rely on pre-exposure tests and an accurate surface up to which the emulsion can be pressed. My own method uses a second microscope mounted behind or beneath the stage which can be used to observe the primary image, as well as the actual emulsion surface. By placing a red filter in the light path it is possible to adjust each slide to ensure that the image and emulsion are in the same plane

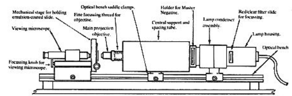

FIG. 1. Microphotographic Projection Apparatus.

without exposing the emulsion. Although the emulsion is normally completely transparent, in practice it will contain minute particles of impurities and the smallest visible of these are chosen for focussing purposes.

The other advantage of this method of focussing is that if the slide is mounted on a mechanical stage then the slide can be moved slightly to select the cleanest part of the emulsion for the microphotograph. Once focussed, the red filter can be removed, thus starting the exposure.

The apparatus, shown in the diagram, was made out of various parts of microscopes, cameras and surplus lenses. The parts being mounted on an optical bench. The principle still follows that described above and indeed the first set-up successfully attempted used a Russian MBR1E microscope with an angled viewing microscope mounted in the substage condenser ring and a 300 mm length of 50 mm plastic water pipe holding the film and illumination source vertically above the objective.

The optical bench version has a 12 v 100 w quartz halogen light source followed by a series of large-aperture government surplus camera lenses giving very even but strong illumination of the 35 mm negative mounted in the film holder. Assuming that a single objective is used to act as the reducing lens, the amount of reduction is equal to the ratio of the distance between the film and the optic centre of the lens and the optic centre of the lens and the emulsion. The first distance is kept relatively large which in turn reduces the angular field of the objective ensuring a flat field. The image is as large as possible on the negative so most of the reduction is carried out on the copying apparatus. This means that higher NA and higher resolution lenses can be used giving greater resolution and shorter exposure times.

The large centre tube of the apparatus does nothing more than act as a spacer and support for the optics. The main reducing objective is mounted on a fine thread used for focussing purposes. The emulsion coated slide is held in a mechanical stage which in turn is fitted to a block that can slide on the optical bench. Also mounted on this block is the viewing microscope, aligned on the optical axis and focussed on the emulsion surface by means of an independant adjustment.

Exposure and development

The reduction ratio, choice of objective and subject matter is a function of individual choice and availability of optics. In general, however, an image size 2 mm to 3 mm square gives a slide that can be easily demonstrated without resorting to elaborate illumination systems. Using projection distances in the order of 250 to 300 mm and objectives in the range 12 mm to 35 mm gives a suitable starting point and assuming a good illumination on the negative the exposure time may vary from 30 seconds to two minutes. Care must be taken to ensure that the emulsion side faces towards the light source and that the focussing telescope is set on the emulsion side of the slide and not the back surface. Once the image focus (in red light) is co-incident with the focus of a minute particle embedded in the emulsion, the red filter can be removed, starting the exposure.

Various developers can be used. Perceptol being the one chosen for the most recent slides produced. The slides are developed using a red light source under a glass developing dish and a low-power stereo microscope to observe the progress of the development. Conventional film fixers are used and the slides dried ready for covering.

In order to give the slides a neat finished appearance the outer edges of the emulsion can be removed using a ringing table and scalpel. The remaining circle of emulsion containing the picture can then be ringed using dry mount adhesive with some black drawing ink added. The final covering being a slip and Canada balsam. The xylene in the balsam can be partially driven off before the slip is lowered by heating the balsam placed on the slide. This accelerates setting after the slip is lowered, and the emulsion is unaffected by the moderate temperatures required.

Future developments

Although microphotography is a subject that should not be considered without careful forethought, it does offer an absorbing project that has not been attempted in recent years by many amateurs. It can involve many hours of interesting experimental work in analysing the problems, and noting the relative influence of various combinations of lighting, objectives, developers and techniques to produce the emulsion.

Some of the first microphotographs were produced by Dancer in the mid-1800s, soon after photography was developed and was popular up until the turn of the century when many slides were made for commercial sale. Today very little is carried out in amateur circles apart from the exhibiting of those earlier Dancer and other microphotographs. It is hoped that these notes may encourage other amateurs to make a serious attempt at a neglected area of microscopy.

REFERENCES

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