

MICRORADIOGRAPHY AND X-RAY MICROSCOPY IN GEOLOGY

W.L. JONGEBLOED¹⁾ and D.H. PORRENGA²⁾

SUMMARY

Photographs have been made of a specimen of *Operculina* with an X-ray projection microscope, a light microscope and by means of microradiography. Comparison of the pictures obtained shows that, if original unsectioned specimens are examined, the two X-ray methods are superior to the light-microscope technique as regards both depth of focus and penetration power. These methods are moreover non-destructive and time saving.

Results obtained also show that for the rapid and routine investigation of microfossils or for detailed study, the microradiography and X-ray microscopy methods are, respectively, equal or superior to the light-microscope technique.

INTRODUCTION

The use of microradiography and X-ray microscopy in geology is not new. The first published pictures of fossils taken by means of X-rays appeared several decades ago, while X-ray photography of sedimentary rock samples has been widely applied since 1962. A direct comparison between microradiography, X-ray microscopy and light-microscopy based on photographs obtained from the same geological specimen has never been published, however. Such a comparison, using a specimen of *Operculina*, has now been made and shows "in a nutshell" both the advantages and the drawbacks of the various methods. This, together with a brief discussion of the principles of microradiography and X-ray microscopy, is the subject of the present paper.

MICRORADIOGRAPHY

Method

In microradiography a specimen mounted on a photographic film or plate is irradiated (fig. 1). Differences in the structure or texture and in the

chemical composition of the specimen cause differences in the absorption of the X-radiation, resulting in a shadow picture of the specimen.

In principle, a variety of X-ray instruments, ranging from medical instruments to industrial diffraction units equipped with various tubes and anode materials, can be used. At KSEPL the use of a portable MACROTANK B was found to be most useful for the photography of cores and sediment slabs, while for the photography of Foraminifera and other (micro-) fossils, a unit for X-ray diffraction was found to give the most satisfactory results.

The 100 kV/4 mA MACROTANK B consists of an X-ray tank, a control panel and cables for the mains connection and for the connection of tank and panel. The oil-filled tank contains the X-ray tube, which has a focal spot of 1.0 x 1.0 mm and the H.T. transformer. The panel contains selectors for setting kV and mA, kV- and mA-meters, and a timer for automatic control of exposure time. The relatively large focal spot and the divergent X-ray beam result in a lower resolution than is obtained in X-ray microscopy. The

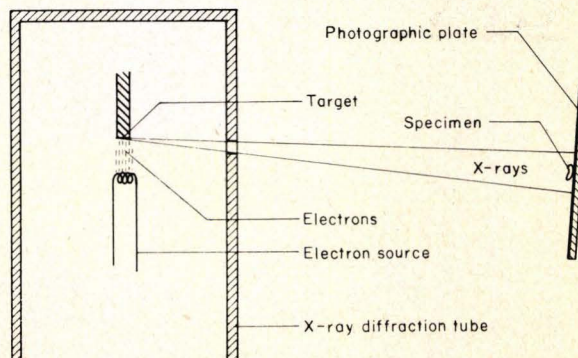


Fig. 1 - Principle of microradiography

¹⁾ Technisch-Fysische Dienst T.N.O. en T.H., Stieltjesweg 1, Delft, The Netherlands

²⁾ Koninklijke/Shell Exploratie en Productie Laboratorium, Rijswijk, The Netherlands

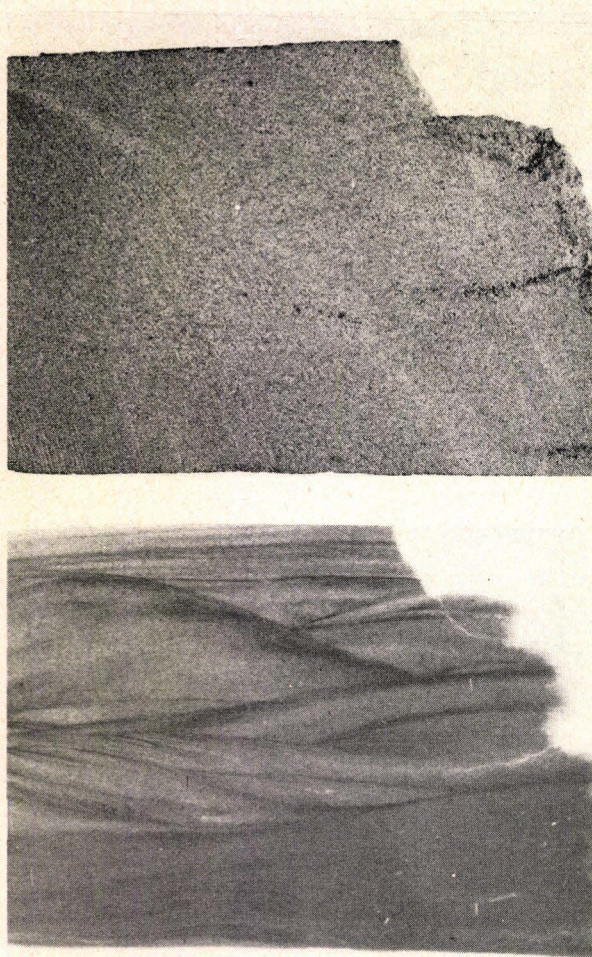


Fig. 2 - Sandstone from the Spinti formation (Oligocene), Scrivia area, Northern Apennines, Italy.

- A. Normal photograph of the rock slab (natural scale) showing saw marks only. No sedimentary structures visible.
- B. X-ray photograph of the same rock slab, showing cross bedding overlain by horizontal lamination.

instrumentation, however, is much simpler and cheaper. In contrast with X-ray microscopy, microradiography permits large objects with a diameter of several decimetres or, simultaneously, a great number of small objects, to be photographed if the distance from the source to the object is kept at 100 to 120 cm.

Results

Sedimentary rocks

Good results are obtained if the sedimentary rock samples are cut perpendicular to the observed or expected bedding plane into flat slabs 2 to 10 mm thick. With operating conditions of 40 kV and 4 mA

an exposure time of 2 to 6 minutes is usually sufficient to obtain pictures of optimum quality (fig. 2). Because many papers on this subject have been published in recent years (Hamblin, 1962; Calvert and Veevers, 1962; Stanley and Blanchard, 1967) there is no need for a lengthy discussion of applications in this paper.

Microfossils

We have replaced the beam-exit tube of a Debye-Scherrer 114.83 mm powder camera by a flat rectangular device on which a piece of Kodak Maximum Resolution plate can be mounted easily. Specimens to be photographed, say a number of Foraminifera, are glued to the paper-wrapped photographic plate. The camera is used without collimators. After 5 or 6 minutes exposure to copy radiation at 36 kV and 20 mA the plate is developed in Agfa Graphic Arts Developer (Varitol Normal) in accordance with the manufacturer's instructions. The ultrafine-grain emulsion permits enlargements to be made of more than 300 times.

This radiographic technique is most suitable for the routine investigation of many small specimens. Depending on their size, up to 100 microfossil

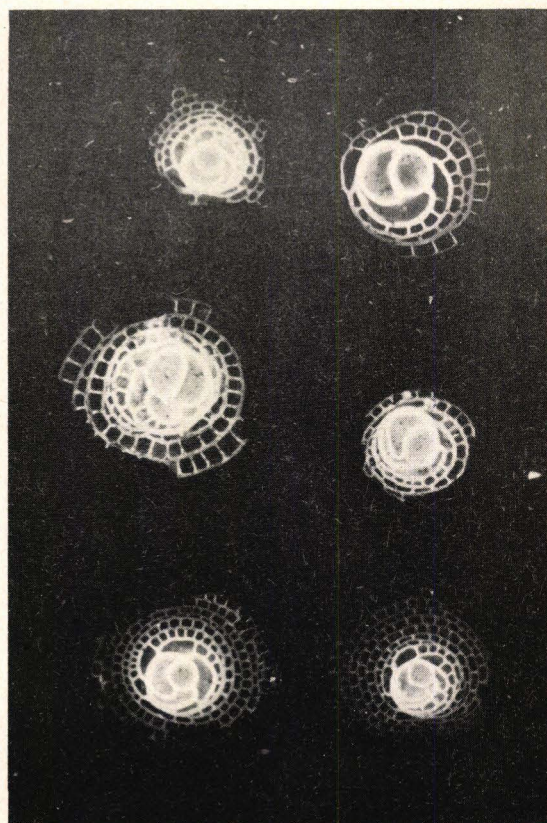


Fig. 3 - Several specimens of *Cycloclypens* photographed by means of microradiography. Magnification 25 x.

specimens can be photographed at the same time. Figure 3 is a picture taken of six different specimens of *Cycloclypeus* and figure 5a shows a microradiograph of a specimen of *Operculina*. In both pictures the outline of the initial chambers is clearly visible, but details of the microstructure are not resolved. The *Operculina* specimen has also been examined with the X-ray microscope and by means of the light microscope (using one half of the specimen, which was split into two equal parts for this purpose). The outcome of this comparison will be discussed below.

X-RAY MICROSCOPY

Method

In the X-ray projection microscope an extremely fine point-shaped X-ray source is obtained by focusing electrons onto a thin metal film (thickness 1-2 micron). The X-ray source irradiates a specimen which is mounted very close to it. An enlarged shadow image of the specimen is obtained on a fluorescent screen or a photographic film or

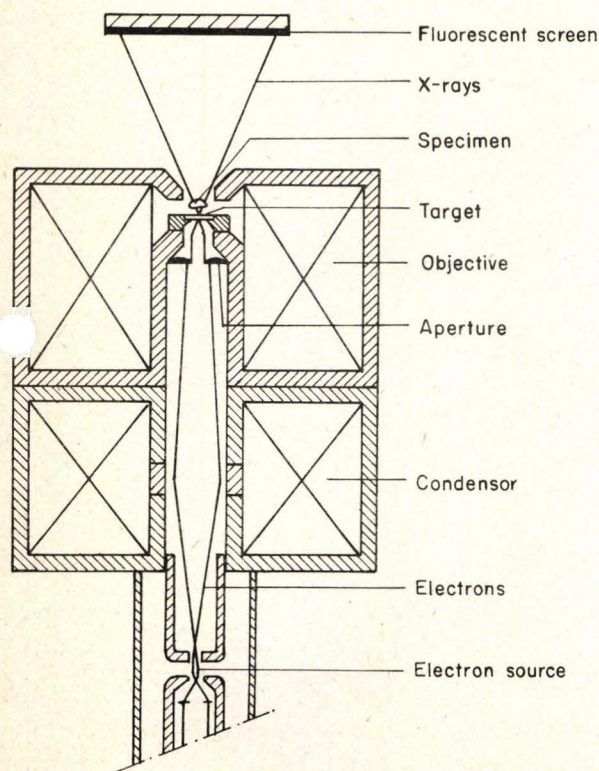


Fig. 4 - Principle of the X-ray projection microscope.

plate, located further away from the source. A diagram of the path of the rays is given in figure 4. All parts featured in it, viz. electron source, two electro-magnetic lenses, target holder, specimen holder and camera or fluorescent screen are mounted in a microscope tube which is evacuated during operation.

The difference between the microradiographic method and the X-ray microscopic method has some important consequences for the resolution and the maximum enlargement of the image (Jongebloed and Jutte, 1965). In X-ray microscopy, the point-shaped X-ray source results in a resolution of about 0.25 micron (Ong Sing Poen, 1961), a scale not approached by microradiography. When an X-ray microscope is used, the initial magnification of the specimen on the film ranges from 10 to 110 times, while in microradiography no initial magnification is obtained. An additional advantage of the X-ray microscope is the ease with which stereoscopic images can be made.

Both methods are superior to light microscopy in that they achieve a great depth of focus, a high penetration power and usually a high contrast in the shadow image. All these points, the importance of which has already been demonstrated in botanical work, paper research and in biomedical studies (Jongebloed, 1965; Salmon, 1956), are equally advantageous in the study of microfossils.

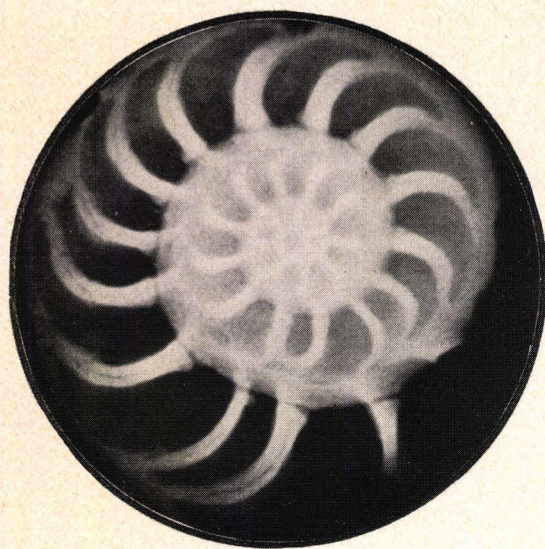
Results

Various X-ray techniques have been employed for the study of microfossils (Hedley, 1957; Hooper, 1960), but a comparison based on the results obtained with one and the same specimen has not been made until now. Figure 5 shows four photographs taken by

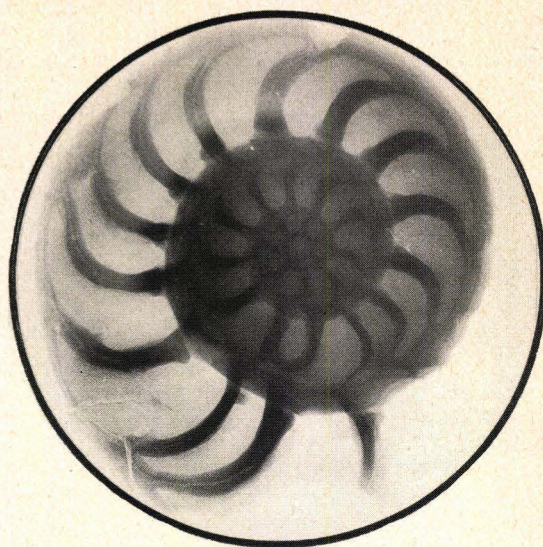
- the microradiography method
- the X-ray microscope
- the light microscope (using one half of the split specimen) and
- the X-ray microscope (also using one half of the split specimen),

the subject being a specimen of *Operculina*.

The pictures obtained with the X-ray microscope show much more detail, especially in the pores and the rim of the outer chambers, than the microradiographic picture, in which only the shell construction is clear. In figure 5c the picture shown was obtained with a common microscope; the specimen is embedded in Lakeside cement. The initial chambers are visible, but the lack of depth of focus is obvious. A drawback of the light microscope technique is that the splitting or sectioning of microfossils is often a tedious and time-consuming work. In figure 5d a picture is shown of the same half of the *Oper-*



a



b

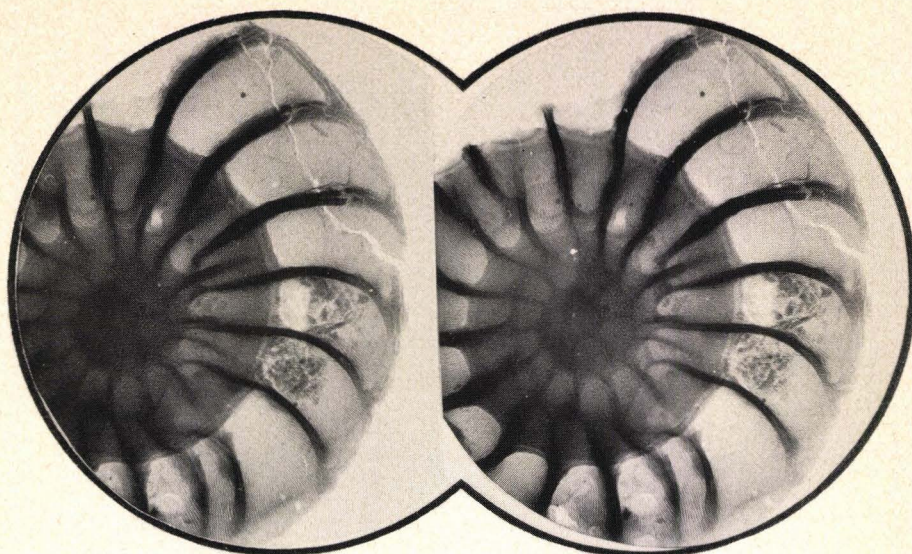


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d

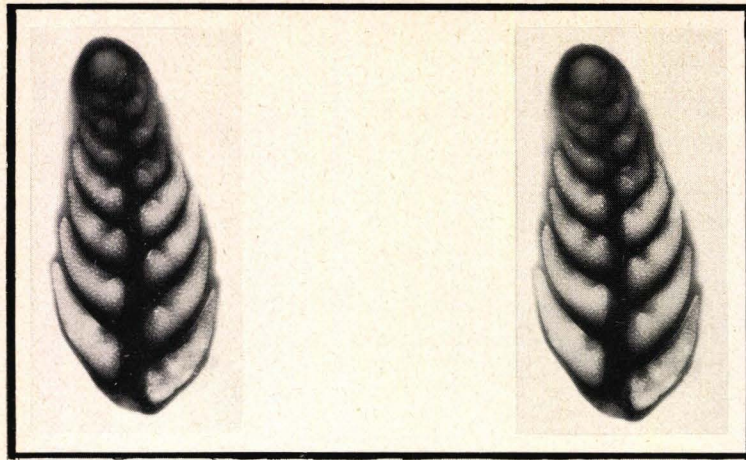
Fig. 5 - *Operculina*, photographed four times by different techniques. Magnification 100 x.
 a) Microradiography. Inner shell construction well revealed. Details not resolved.
 b) X-ray microscopy. Details of the shell construction are well resolved.
 c) Light microscopy. One half of the split specimen, in reflected light. The initial chambers are clearly visible, but the insufficient depth of focus gives rise to considerable lack of sharpness in the image of the other parts of the specimen.
 d) X-ray microscopy. Same section as that used for 5c. The cement used for 5c has been dissolved in alcohol. Chambers and pores are well resolved.



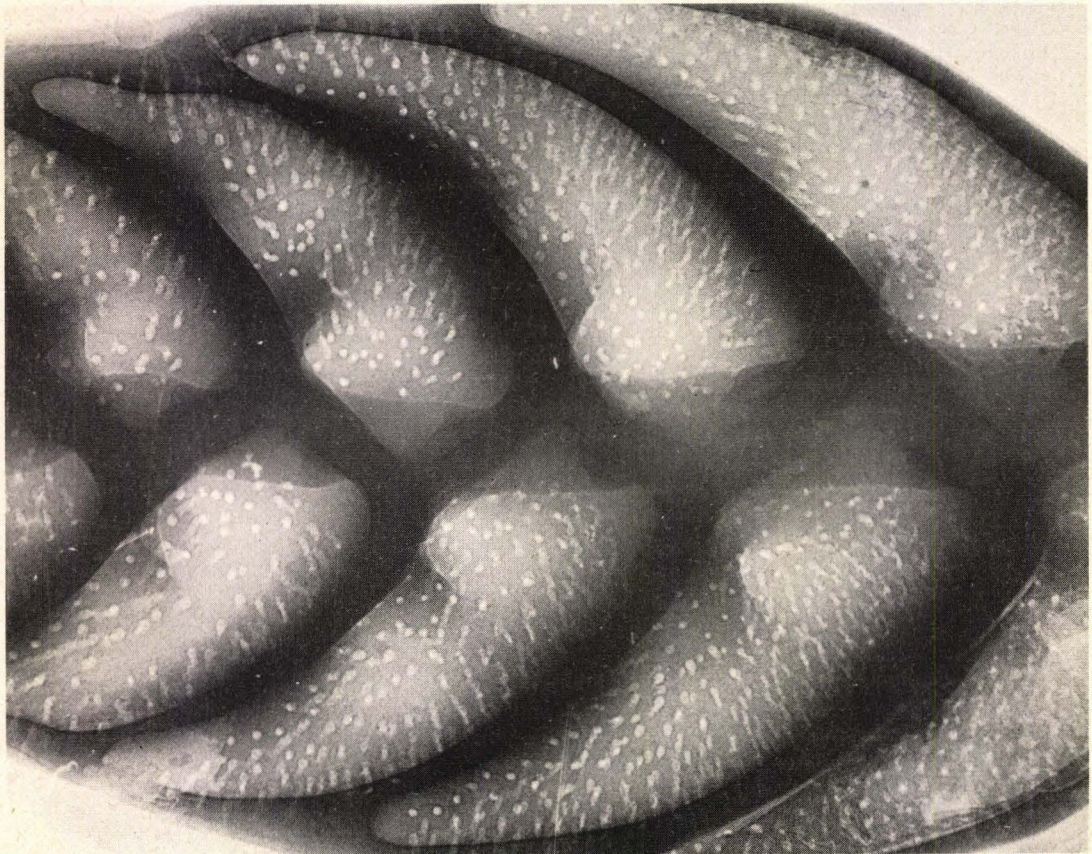
a



Fig. 6 - a) Stereoscopic image of a part of *Operculina*, taken by means of the X-ray microscope. The arrangement of chambers can be studied. For the study of pore size and number of pores, a detail enlargement as shown in the lower figure b) is more suitable.



a



b

Fig. 7 - a) Stereoscopic image of *Bolivina* taken with the X-ray microscope, showing the spatial arrangement of chambers. Magnification 120 x.
b) Enlarged detail of *Bolivina* for investigation of pores. Magnification about 600 x.

culina specimen as that used for picture 5c, the cement now having been removed to increase the contrast, however. Again, the chambers and pores are clearly revealed.

The stereoscopic image of the specimen, or part of it, reveals more information on the spatial structure than the picture in figure 5. Figure 6 is an enlargement of a detail of the body of an *Operculina* specimen. Pore concentration and pore diameter can be examined. Similar results have been obtained with a specimen of *Bolivina*, the stereoscopic image of which is given in figure 7a. An enlarged detail, showing the distribution of pores, is given in figure 7b. The various shapes of the pore images (circles at the centre, changing to ellipses and lines at the periphery) show that a projection method has been applied.

CONCLUSIONS

Because of the great depth of focus of the picture and the high penetration power of the X-rays, the two X-ray methods give better results than the light microscope if thicker specimens have to be examined. Usually neither splitting nor sectioning of the specimens is necessary. Both X-ray methods are therefore generally non-destructive and time saving.

For rapid and routine examination of microfossils the microradiography method is to be preferred. The X-ray microscope method is preferable when structural details such as pore diameters, pore concentration, shape and implantation of spines, thickness of the rim, have to be investigated.

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