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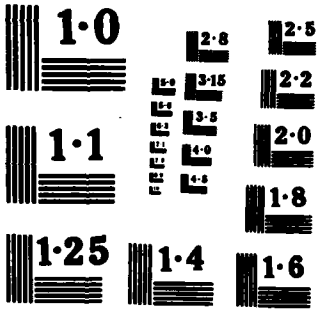
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ROYAL AIRCRAFT ESTABLISHMENT

ON THE OPTICAL ASSESSMENT OF THE VOID CONTENT IN COMPOSITE MATERIALS

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SUMMARY

Two simple optical techniques for the determination of the void content in composite materials are described. The nature of voids is discussed and a practical classification proposed. On the basis of this it is suggested that the techniques described are more than adequate for the assessment of material quality for both development and production purposes. Notes on the techniques employed to obtain a satisfactory polished section are appended.

Additional keywords: Great Britain; fibre epoxy composites; volume fraction.

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1 INTRODUCTION

In order to make full use of composite materials in aircraft structures it is necessary to be able to assess the quality of the material simply and quickly both for design purposes and as a means of ensuring the employment of satisfactory manufacturing techniques. One such quality measurement required is the composite void content. Acid digestion techniques give reliable measurements of void content of fibre-resin composites although only if expertly carried out and if the component densities are accurately known. However, the techniques are not applicable to all types of fibre composites and are not simple to execute. In addition, the densities in the manufactured composite may not be accurately known. These difficulties are reflected in the frequent quotation of negative void contents.

This document describes two simple techniques for the assessment of the void content in a composite using standard low power optical microscopy. → See p1

2 THE NATURE OF VOIDS IN FIBRE-EPOXY COMPOSITES

The measurement of void content must of necessity be a mean value taken through a given volume or over a given cross-sectional area.

It is clear therefore that simple void content quotations have significance only for fairly uniformly distributed voids. To quote a void content of, say, 0.5% for a composite of generally uniform high quality ($V < 0.2\%$) but containing an occasional very large void would be misleading and indeed could be dangerous. It is suggested that for this case the statement of void content should read "generally $0 < V < 0.2\%$; infrequent, local $V > 5.0\%$ " (see Fig 1). The following discussion therefore refers to the void contents for composites in which the voids are fairly uniformly distributed.

Optical and scanning electron microscopy of a variety of fibre composites has shown that the type of void encountered is a function of the manufacturing technique and varies with the void content. Since, apart from hollow fibres, voids can only occur in the resin, they tend to be found in resin rich areas which occur between tows (usually in filament wound material) or between laminae. If a section of a unidirectional composite is taken in a plane perpendicular to the fibres, the following generalised classification of voids may be made:

(a) In high quality composites where $V < 0.5\%$, voids are usually caused by entrapped volatiles. In section they appear circular, having a diameter in the order of 10 μm.

(b) As V approaches 1%, voids of the type described in (a) tend to decrease in number and are replaced by (or combined with) larger pockets usually appearing between tows or laminae. In laminated material these pockets frequently occur in the vicinity of a misaligned fibre where laminate compaction is restricted. They thus tend to be about one fibre diameter in thickness and in varying lengths up to 100 μm.

(c) As V increases beyond 1%, the voids occur almost exclusively as those in (b) but increase not only in frequency but also in size. A useful rough guide is that at $V = 2\%$ the average void thickness $\triangleq$ 2 fibre diameters, and at $V = 5\%$ the average void thickness >5 fibre diameters.

Even within the relatively small area of a microscope sample, the void content may vary considerably, particularly in poor quality laminates. Thus it is more meaningful to quote void contents as lying within a given band than to state a specific average figure.

Following from this discussion of the nature of the voids it seems reasonable to suggest that sufficient information for most purposes would be provided by quoting the void contents as lying within one of the following six groups:-

$$V < 0.2\%$$
$$0.2\% < V < 0.5\%$$
$$0.5\% < V < 1.0\%$$
$$1.0\% < V < 2.0\%$$
$$2.0\% < V < 5.0\%$$
$$V > 5.0\%$$

The above descriptions of the nature of the voids refers only to a section of unidirectional composite taken perpendicular to the fibres. Because of the tendency of voids to occur in resin rich areas, sections of a laminated composite taken parallel to the laminae would be likely grossly to over- or under-estimate the void content depending, respectively, on whether the section occurred at an interface between laminae or elsewhere. This should be borne in mind for angle-ply composites but adequate accuracy should be obtained by ensuring that any winding or laminating planes are perpendicular to the plane of the section to be considered and that all fibre orientations are at an angle to that plane.

3 SPECIMEN PREPARATION

The sample to be analysed should, when possible, be of the total thickness of the composite and, for the mounting techniques currently used, approximately 15 mm square. Standard optical microscopy mounting and polishing techniques (see Appendix) for fibre composites were used to prepare the specimens discussed in the next section. The polished finish need only be sufficient to enable the void dimensions to be judged to an accuracy of about 10%.

4 OPTICAL COMPARISON TECHNIQUE

Illustrated in Fig 2 are typical void distributions in a laminated composite drawn for 0.2%, 0.5%, 1.0%, 2.0% and 5.0% void content. Each figure shows the appearance of the voids at ×40 magnification. By using an optical microscope at magnifications of the order shown on the figures it is possible to arrive at an estimation of the void content of a composite by simple comparison between Fig 2 and the microscopic view of the section. Such an estimation is extremely quick to make and of sufficient accuracy for most applications. This visual technique also allows an assessment of the types and distribution of voids to be made.

Use of a 10 × 10 squared graticule in the eyepiece, as described in the ensuing paragraph, may assist in the assement. A typical carbon fibre composite section is illustrated in Fig 3 with the effect of an eyepiece squared graticule superimposed.

First, note that the voids are in general between 1 and 2 fibre diameters in thickness and of the order of 100 μm long (fibre diameter ≙ 8 μm) and that there are no volatile voids. By comparison with Fig 2 this immediately places the void content between 1% and 5%. Using the graticule, each void within the 10 × 10 area is assessed as a fraction of the area of a small square and the total void area within the graticule as a function of the area of a small square determined. It is easy to establish that, in the case illustrated, the voids occupy in total about two small squares in area. Hence the average void content over the complete area considered is determined as 2%. A mean void content determined from several such sections would of course provide greater accuracy over a larger and hence more representative area.

5 OPTICAL COUNTING TECHNIQUE

An alternative way of assessing the void content is to sample the area in a regular pattern and simply to note the presence or absence of a void. Consider a 10 × 100 square grid covering a representative area of composite at a magnification such that the voids are of the order 0.1 to 1.0 × the area of each small square, as in the Optical Comparison Technique. By counting the number of times the precise centre of any small square lies over some part of a void a statistical assessment of the void content may be made. Take for example the case of a composite having a void content of exactly 2%; the probability is that the precise centre of a small square will be over some part of a void 20 times (10 × 100 × 0.2). To accomplish such an assessment in practice using an eyepiece having a 10 × 100 graticule would be difficult due to the necessary reduction in graticule size. However, if the centres of the 10 squares across the width are represented by ten marks on a vertical line in the eyepiece and the microscope stage adjusted so that the line is moved horizontally 99 times at intervals of one square width, the effect of a 10 × 100 graticule will be obtained. If then, at each of the 100 positions of the line the number of times a part of a void falls on one of the ten marks is recorded, the total obtained from the 100 recordings will give a measure of 10 V%. Because, particularly in laminated composites, we have seen that voids tend to occur in lines along lamina boundaries, it is necessary to ensure that the 'horizontal' movement of the microscope stage is such that the 'vertical' graticule line crosses a representative number of laminae, *ie* the laminate plane is at an angle to the 'horizon' (Fig 4). It should also be noted that the horizontal intervals may be increased by a factor of 2 or so to cover a greater area, without affecting the accuracy significantly, although the finer the grid the greater the accuracy. Fig 4 shows a typical carbon fibre composite with a 10 × 100 traverse indicated - in practice the positions marked as dots would be fine crossed lines in the eyepieces.

It should be noted that this technique may also be used to assess the composite fibre content if similar conditions are observed, *ie* the section is perpendicular to the fibres, the fibre diameter is of the order of 0.1 grid interval and the traverse is made at an angle to the laminae.

6 CONCLUSIONS

It is suggested that for most purposes it is sufficient to quote void contents in the following bands:

Grade A	-	$V < 0.2\%$	-	Excellent
Grade B	-	$0.2\% < V < 0.5\%$	-	Very good
Grade C	-	$0.5\% < V < 1.0\%$	-	Good
Grade D	-	$1.0\% < V < 2.0\%$	-	Fair
Grade E	-	$2.0\% < V < 5.0\%$	-	Poor
Grade F	-	$V > 5.0\%$	-	Very poor.

Two simple optical techniques have been described which should be adequate to characterise the void content of most fibre composites to the above accuracy. The second technique may also be used to assess fibre content. Limited experimental use of the techniques suggests that sufficient accuracy is obtained in practice.

Appendix

PREPARATION OF SPECIMENS FOR MICROSCOPIC EXAMINATION

A.1 Specimen mounting

To facilitate the polishing process, the specimen is cast in a polishing medium - normally glass-filled diallyl phthalate. This provides a hard, easily-polished surround to the specimen. However, when the temperature and pressure required to cure the above medium are likely to affect the specimen, a cold-setting epoxy resin* is used.

A.1.1 Diallyl phthalate

The mould used for this polishing medium is shown in Fig 5. The brass base B and plunger P are good sliding fits at room temperature in the 30 mm inside diameter steel cylinder C . The specimen, the maximum dimension of which should not exceed 20 mm, is positioned with the face to be polished resting on the base and the cylinder fitted. Taking care not to disturb the specimen, diallyl phthalate powder is poured into the cylinder to a level 5 mm below its tip. The plunger P is then inserted as illustrated in Fig 5. The complete mould is placed between the platens of a temperature controlled press and a constant load of 1000 kgf applied; the platen temperature is set to 150°C and the heaters switched on. The heaters are switched off 30 minutes after the platens reach 150°C. The mould is allowed to cool under pressure until the temperature has dropped below 50°C before removing the mould. No attempt should be made to separate the mould until it has reached room temperature since the differential thermal expansion of the brass and steel provide a tight seal at elevated temperatures.

A.1.2 Epoxy resin

Approximately 10 ml of resin* are prepared. The specimen is stood on the removable base of a standard re-usable plastic mould** and the resin poured around and over the specimen to fill the mould. The resin is allowed to cure at room temperature for 24 hours.

A.2 Polishing technique

A.2.1 Initial preparation

The centre of the reverse side of the specimen is first jig-drilled to provide a 5mm deep, 4.6mm diameter hole. The obverse side is then prepared by hand under running water on a series of abrasive papers from grade 280, 400, 600-1200 until the surface is free of major defects and the fibres are clearly visible under a low-power microscope. From this stage onwards extreme cleanliness is necessary and the specimen should be ultrasonically cleaned at each stage. Ultrasonic cleaning between the use of successive grades of abrasive paper is also advisable.

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* Ciba Geigy 753/956 80:20
**Metaserv 30 mm, Metallurgical Services, Betchworth

Fig 2

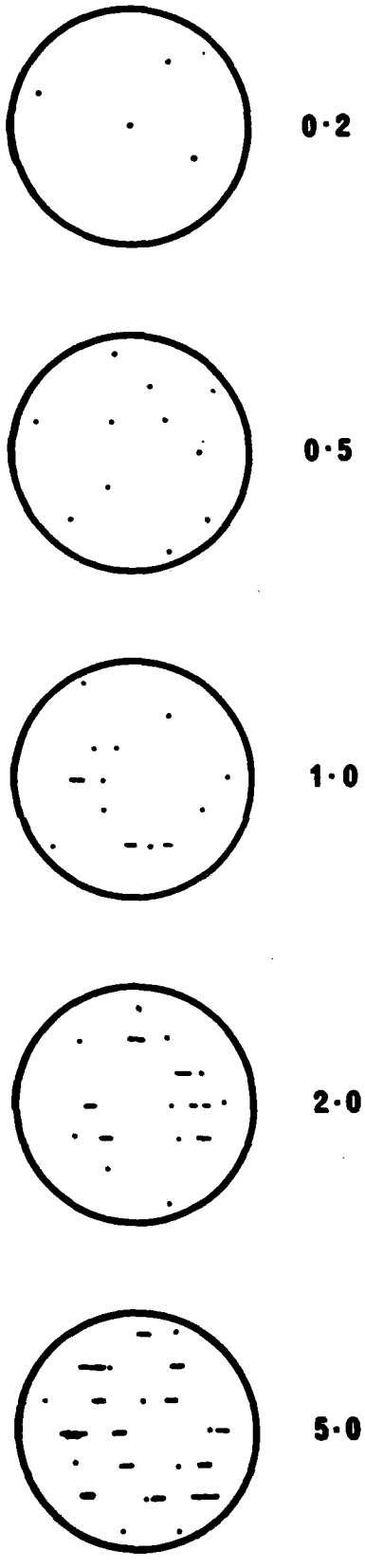

Fig 2 Void type and distribution at V = 0.2%, 0.5%, 1.0%, 2.0%, 5.0% (x40)

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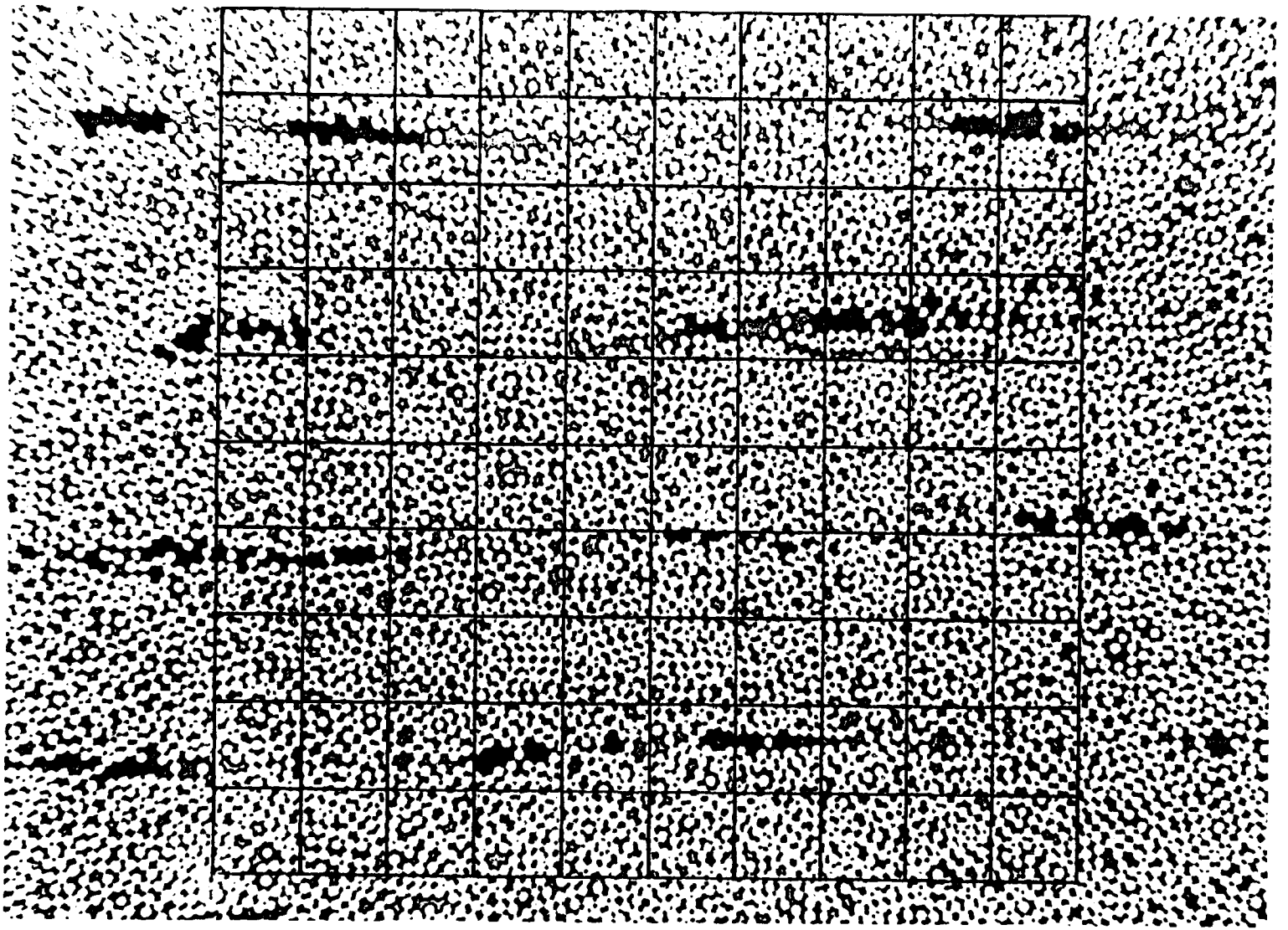
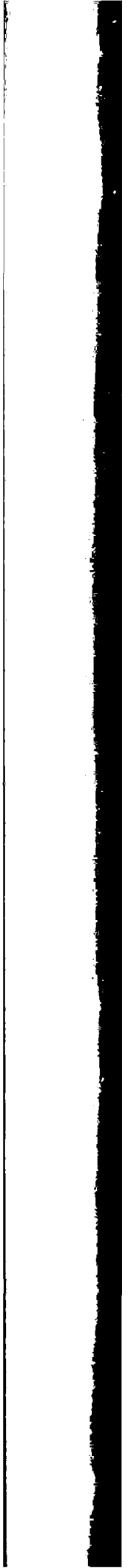


Fig 3 2% void content composite with 10 x 10 graticule superimposed (x300)

Fig 3



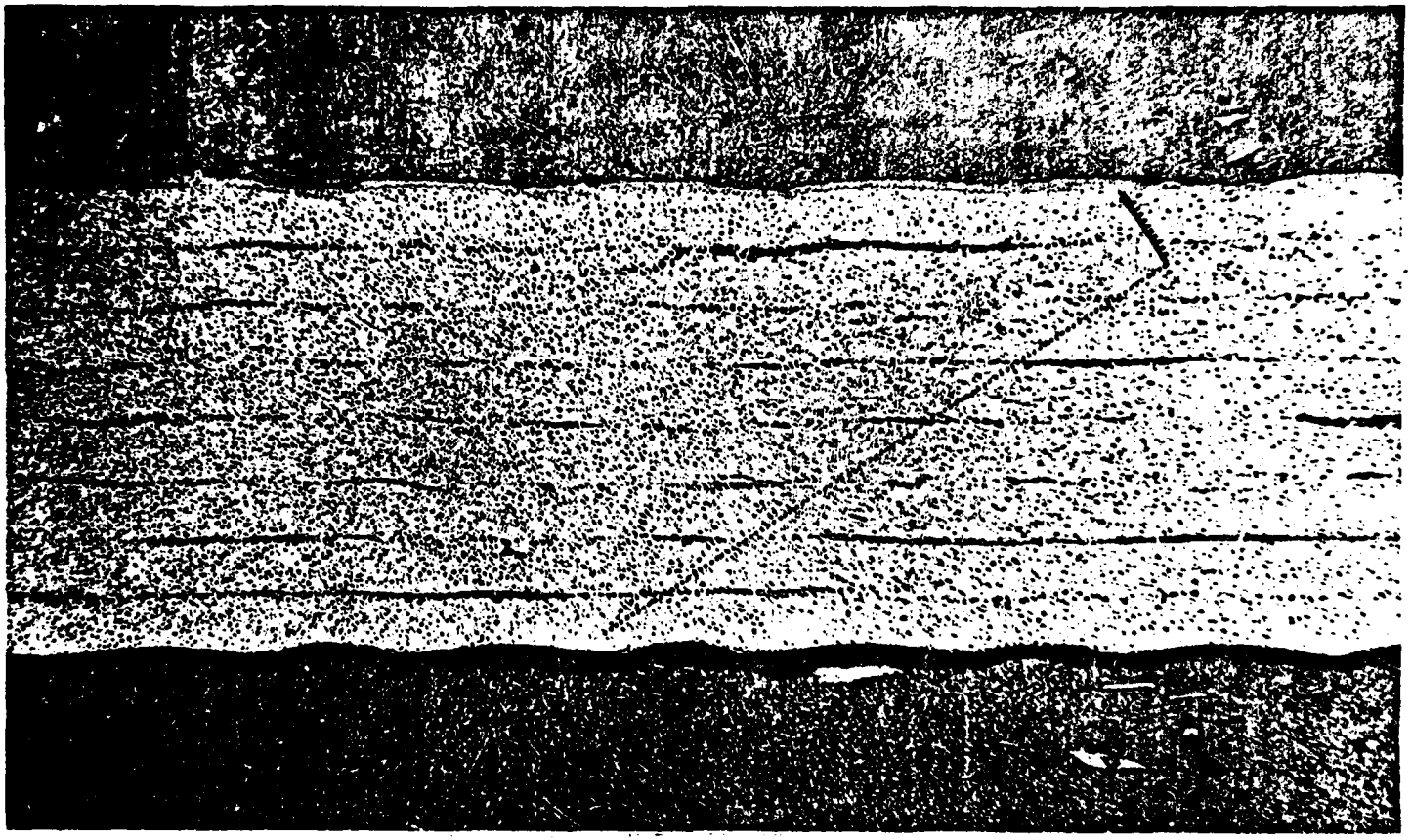


Fig 4 Composite section showing the 100 positions of a 10 x 100 traverse (x70)

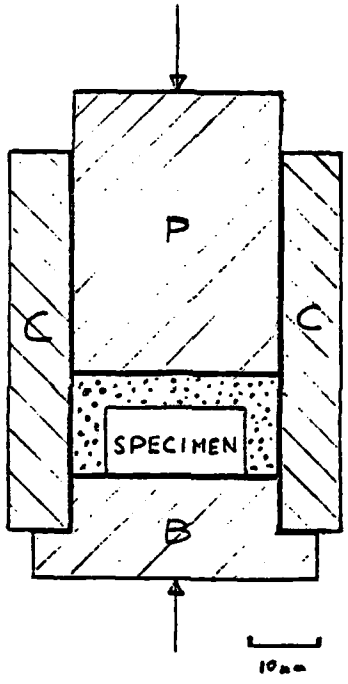


Fig 5 High temperature mould [scale 1:1]

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17. Abstract

Two simple optical techniques for the determination of the void content in composite materials are described. The nature of voids is discussed and a practical classification proposed. On the basis of this it is suggested that the techniques described are more than adequate for the assessment of material quality for both development and production purposes. Notes on the techniques employed to obtain a satisfactory polished section are appended.

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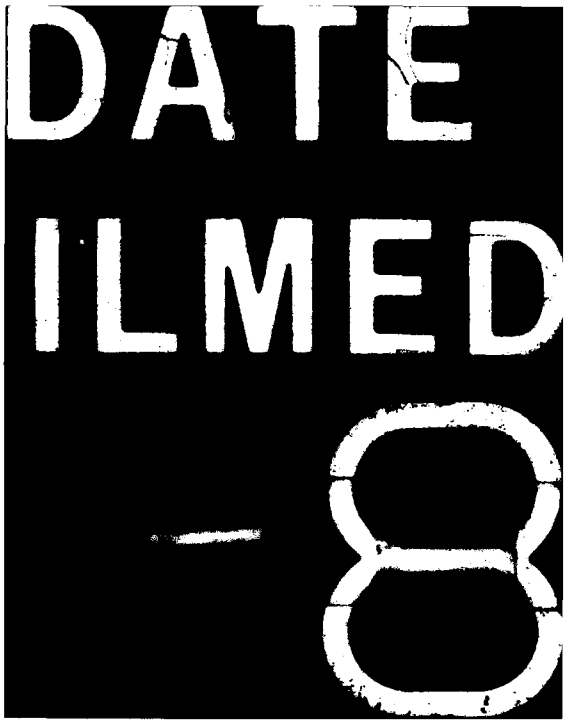
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