

## TECHNIQUES FOR THE DETERMINATION OF COMPLETE POLEFIGURES USING COMPOSITE SPECIMENS

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*Abstract:* A simple, accurate and flexible method is described for the preparation of composite samples from metal sheets. The basic technique can be adapted to conserve material or for the measurement of thick plate specimens. Any combination of the four possible quadrants of the polefigure can be determined singly or combined. In its routine application the technique is ideally suited to the measurement of three complete polefigures for the determination of orientation distribution functions. In particular, the through thickness integration, sheet area sampling and applicability to large grained materials make it suitable for the measurement of texture for comparison with bulk physical properties. Complete instructions are given for the application of the technique in practice.

### INTRODUCTION

Several methods have been described for preparing one quadrant of an X-ray polefigure by composite sampling techniques.<sup>1-6</sup> These are all based on a specimen in sheet form which is cut and stacked to form a block which is subsequently machined. Little or no correction for defocussing or absorption is required, and the preferred orientations are integrated through the sheet thickness and over a large area of the sheet, thus making the technique especially suitable for the comparison of measured textures and physical properties and materials with a large grain size. The prepared specimen is measured only in back reflection using the Schultz technique.<sup>7</sup> The published methods all suffer drawbacks in practice. The techniques of Lopata and Kula<sup>4</sup> and Meieran<sup>3</sup> require very complex, and potentially inexact, machining operations to prepare a thin texture specimen such as required in many texture goniometers. The technique of Leber<sup>5</sup> while using very little material suffers a consequent

loss of accuracy and the integration over a wide area of the sheet. The method of Elias and Heckler<sup>6</sup> appears simple to use, but it requires very precise preparation of the coupons in order that they should remain flat when the specimen is cut. A method has been developed from those published which combines simplicity of approach with extreme accuracy if required. The technique is flexible with regard to specimen dimensions and form and an extension of the method enables sampling using only small volumes of material (but with consequent decreased accuracy). The method has been used in the analysis of textures using Orientation Distribution Functions (ODF's) and has been applied to a wide range of materials and specimen geometries.

The need in texture studies for an average both through the thickness and over a large area of the sheet is clear. The apparent ease of texture measurements using a selected sample plane normal to the rolled sheet and modern incomplete pole figure ODF methods leads many researchers to ignore the possibly large variations in texture with depth and position. In particular any comparison of measured physical properties with the measured central texture of the material alone is at best speculative; in rolled materials the greatest variation in both texture and microstructure is experienced as a function of depth. Apart from Neutron Diffraction, which is a volume diffraction process, a composite sampling X-ray method is the only reliable approximation to the bulk texture in the sample.

#### TECHNIQUE OF THIN SHEET SAMPLES

The basic technique is summarised in Figure 1. The sheet is marked into coupons of equal width at  $45^\circ$  to the rolling direction. The individual coupons should be clearly marked in a way which enables later re-assembly of the cut sheet. It is of the highest importance that the angles  $\alpha$  and  $\alpha'$ , for all the coupons, are as near identical as possible; differences in these angles cannot be compensated for in later stages. This necessarily requires that the coupons have parallel edges and that the rolled material has a well-defined rolling direction. Any width of the coupons can be accommodated in the simple glueing jig described but for most X-ray goniometers a width of 30 mm is suitable. Sufficient coupons must be cut to give a block of approximately 25 mm thick after glueing. A power guillotine is the most useful tool for cutting the coupons for several reasons. The deviation of successive cuts from parallel with a well-sharpened blade and using a back stop, can be less than .01 mm in a length of 100 mm. This gives a deviation in the important angles  $\alpha/\alpha'$  of .02°. A protractor attachment, if available, can be used for cutting the two halves of the sheet aligning the cut centre line along the protractor in each case. The protractor should not be moved between the two sets of cuts. This method gives a very low deviation between  $\alpha$  and  $\alpha'$  as well as high consistency within the two angles. The power guillotine produces very little extra deformation in the coupons if they

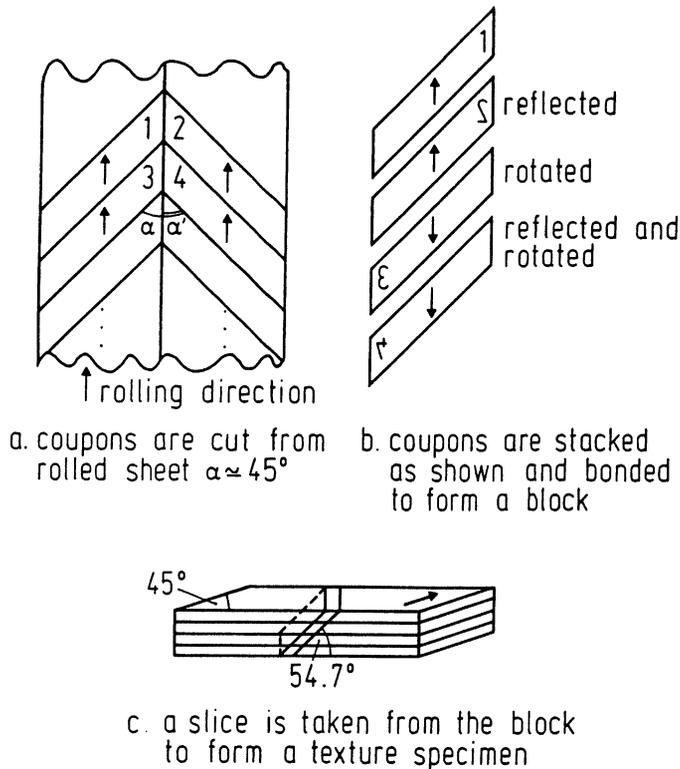


Figure 1. Basic composite specimen technique for sheet material.

are less than 2 mm in thickness. Any deformation which is induced is confined to the edges and will not be sample by subsequent X-ray measurement.

After cutting, the coupons are descaled if necessary, degreased and etched lightly to give a suitable "key" for the adhesive. Suitable cleaning procedures are given by the manufacturers of adhesives but mechanical abrasion should be omitted if possible, particularly where the material is soft or mechanical twinning is possible. For investigation of specifically central regions of the specimens a chemical polish can be used at this stage to remove as much surface material as desired. The coupons are washed and dried before glueing. For most ODF procedures orthotropic sheet symmetry is assumed in the later calculation. The coupons are therefore stacked to form a block, as shown in Figure 1, to average over all four quadrants of the sheet. It is of course possible to change the sampling method so that any of the quadrants are represented separately or combined. A preliminary investigation using incomplete polefigures will establish the symmetry present within the material.

The coupons are glued together to form a block. A custom-built jig is shown in Figure 2, but a length of stock angle iron can serve equally well. The jig is coated with a P.T.F.E. spray release agent and the coupons are coated with adhesive and lain in the jig in the same sequence as stacked.

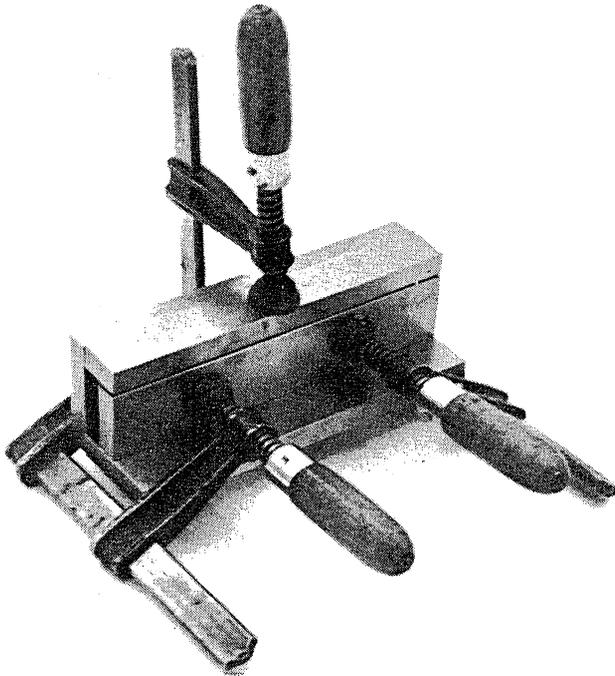


Figure 2. Specially prepared jig used to secure coupons for glueing.

Araldite AW 116 adhesive and HV 953U hardener have been used by the author with much success. The mixed adhesive has good adhesion, is fluid enough to apply by brush, is resistant to shocks when dry and is sufficiently transparent to X-rays. A short pre-cure of the mixed adhesive before application (about 3/4 of an hour at room temperature) is advisable. The glued block is clamped in both directions using standard "G clamps"; it is very important to ensure that the coupons are glued so that they are parallel. The jig takes some practice in order to achieve consistency but it is so flexible in use that it is worth developing the skill; any number of coupons of any width can be glued simply and quickly. The assembly can be heated in an oven to as high as 120°C to speed curing if no texture changes are likely at the elevated temperature. The assembly should be allowed to cool before loosening the clamps. Suitable properties are also obtained in a 24-hour period at room temperature. The block is removed after drying and the faces are ground on carborundum paper to remove surplus adhesive.

The next stage is to cut a slice from the block at 90° to the length of the block and 54.7° to the plane of the coupons. Deviations from both these angles can be rectified in the analysis of the data by computer. Three methods have been successfully applied: spark machining, power abrasive cut-off discs, and hand hack-sawing. A choice must be made according to such possibilities as twinning or recrystallisation during the cut and delamination of the composite block

caused by a too severe cutting stress. A usual thickness for the final specimen is 5 mm. A scrap cut is first made and the specimen is then cut from the centre region of the block. A jig specially made for manual hack-sawing is shown in Figure 3. This technique is most applicable where the mechanical methods are not available or experience has shown that the specimens are liable to break up on cutting. A routine accuracy of  $\pm 1^\circ$  can be achieved.

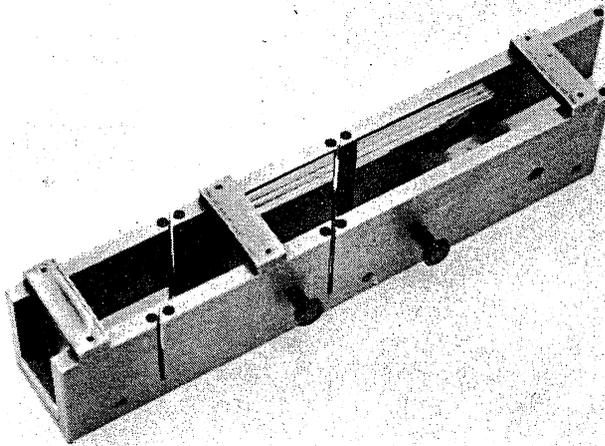


Figure 3. Jig used to hold composite block for hack-sawing by hand.

There are two possible orientations of the block for cutting, leading to two possible orientations of the final specimen. These give right or left handed axial systems when referred to the sheet reference axes. In order to maintain left handed cartesian axes, as usually used in polefigures, the block should be cut in the orientation given in Figure 4.

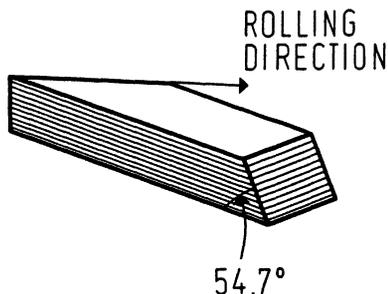


Figure 4. Block assembled from coupons, cut at  $54.7^\circ$ , showing a positive specimen face.

This will be referred to as a positive specimen. The data from a negative specimen can be changed to that from a positive one by inverting the second data index in the data matrix referred to a polar grid based on the sheet reference

axes. It is important in the subsequent ODF calculation that all the data are consistently positive or negative. A completed specimen is shown in Figure 5, showing the sheet reference axes.

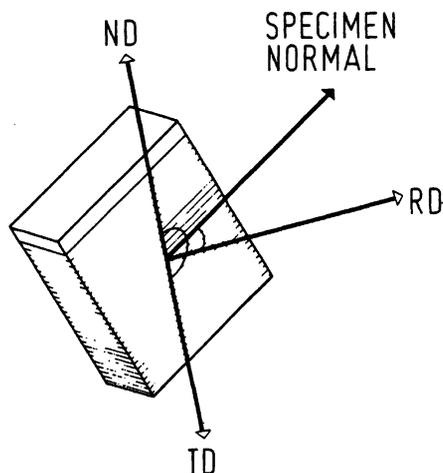


Figure 5. Finished specimen showing L.H. material reference axes for positive specimen.

If the specimens are fragile or need much further machining it is often useful to glue a flat piece of sheet steel of the correct shape to the back of the specimen with quick-drying epoxy-resin adhesive. If a steel plate is used with non-ferrous metals a strong magnet can be used to grip the specimen for grinding and polishing. The specimen is ground on successive grades of carborundum paper to remove the worked layer; this is not usually necessary where the surface has been spark machined. This is followed by some form of polishing procedure to remove the layers damaged by grinding. Chemical polishing is preferable to mechanical polishing and lists of suitable reagents are given in metallographic texts. Care must be taken to avoid reagents which preferentially remove one texture component.

#### DATA ANALYSIS

The specimen is measured by X-rays of suitable wavelength in reflection, data for the reflected intensity being referred to a spiral or polar grid based on the specimen normal. These data usually require mapping onto a polar grid based on the sheet reference axes before being plotted or used for subsequent ODF analysis. Stereographic mapping equations are given by Lopata and Kula<sup>6</sup> but these are unnecessarily cumbersome and those derived by Kallend<sup>8</sup> are to be preferred. By measuring the polefigure, when plotted centred on the specimen normal, we can find the true relationship between the specimen normal and the sheet reference axes. Ideally this should be described by a polar angle of  $54.7^\circ$  with an azimuthal angle

of  $45^\circ$ . The position of the sheet reference axes on the polefigure can be identified by their coincidence with the symmetry axes. (See Figure 6a.) All errors in specimen preparation which lead only to a displacement of the specimen normal

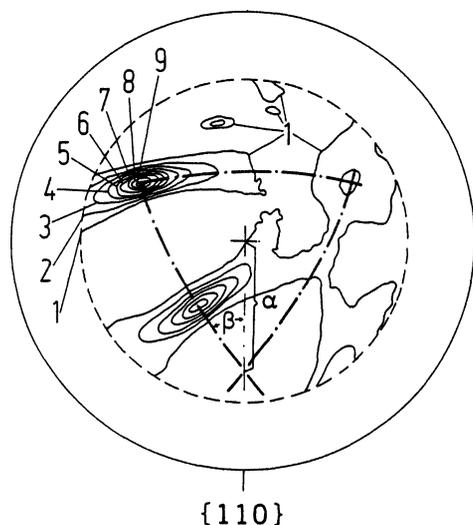


Figure 6a.  $\{110\}$  polefigure for Armco-iron cold-rolled 90%, as measured from composite specimen (normalised over measured region), showing material reference axes and the angles  $\alpha$  and  $\beta$ .

can be corrected at this stage; errors which lead to a relative displacement of the individual coupons cannot; hence the importance of the early stages of preparation.

An interpolation procedure must now be used to map the offset data onto a polar grid based on the original sheet normal. Linear or weighted interpolation procedures have both been used. In the author's case, these have been based on the identification of the four nearest grid points on the measured spiral/net and cross-wise interpolation from these points to the point on the required net. Linear interpolation has been found to be quite satisfactory for subsequent ODF calculations. The possible rounding error where a peak falls between two points is small compared to the truncation errors involved in later ODF analysis. Other corrections, such as for defocussing, dead-time, background can be introduced at this stage where found necessary. The final complete polefigures can then be used as they stand or as input to an ODF routine.

## RESULTS AND DISCUSSION

The author has had direct experience of several thousand satisfactory polefigures being measured by the technique. For the present work however an Armco-iron sample was specially cold-rolled 90% for investigation. Classical

transmission/reflection X-ray measurements (T/R) were undertaken from the central plane of the sheet and a composite sample was also made (without any *abnormal* attention to accuracy). Five polefigures were measured from the composite sample and the polar coordinates of the centre of the polefigure with respect to the sheet reference axes were determined in each case from a 500 mm diameter polefigure similar to that given in Figure 6a. These angles are tabulated in Table I. The polar angle of the centre was relatively constant, lying within a spread of  $1.3^\circ$ . The azimuthal angle showed somewhat more divergence; this can be attributed to a rotation about the specimen normal between measurements in the texture goniometer.

TABLE I

Polar and Azimuthal Angles of the True Centre of the Measured Polefigures (Theoretically  $54.7^\circ$  and  $45.0^\circ$ )

| <u>Polefigure</u> | <u>Polar angle <math>\alpha</math></u> | <u>Azimuthal angle <math>\beta</math></u> |
|-------------------|--|---|
| {100}             | $55.0^\circ$                           | $43.3^\circ$                              |
| {211}             | $55.3^\circ$                           | $42.7^\circ$                              |
| {110}             | $55.8^\circ$                           | $44.0^\circ$                              |
| {310}             | $56.2^\circ$                           | $42.3^\circ$                              |
| {222}             | $56.3^\circ$                           | $44.9^\circ$                              |

A comparison of the interpolated polefigure in Figure 6b with that of the T/R specimen in Figure 6c shows no significant difference. This was observed for subsequent ODF analyses. Differences could have arisen from the comparatively small volume of material, confined to the centre of the sheet, which was sampled by the T/R method. Errors involved in the interpolation procedure are small compared to the errors inherent in other methods, in particular the errors involved in fitting the overlap region in T/R techniques and those inherent in any form of incomplete polefigure measurement. Errors in preparation of the specimen can be kept to a level considerably below those involved in measurement and can mostly be corrected for at a later stage. The variation in diffracting power resulting from the adhesive layer was considered undesirable by Lopata and Kula.<sup>4</sup> This is not a real problem as the complete polefigure is easily normalised and differences arising from different sheet thicknesses can be eliminated. The glue layers are typically less than .01 mm thick in a correctly made specimen and are almost transparent to X-rays. Indeed the absence of the glue in the method of Lopata and Kula can lead to problems in specimen preparation with preferential chemical polishing of the sheet edges and reagent entrapment in the gaps.

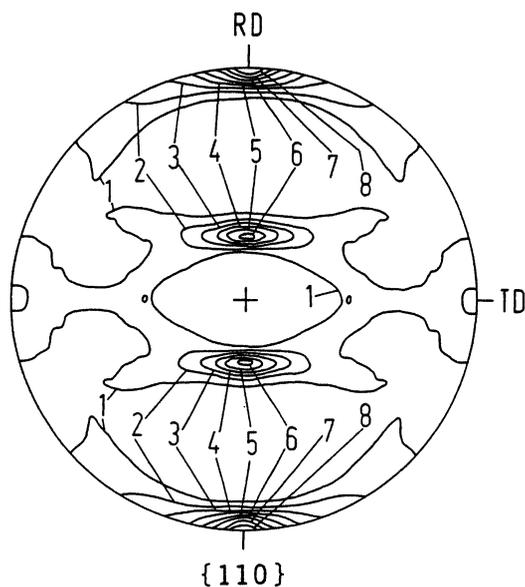


Figure 6b. Same polefigure as in Figure 6a after interpolation and centred on sheet normal.

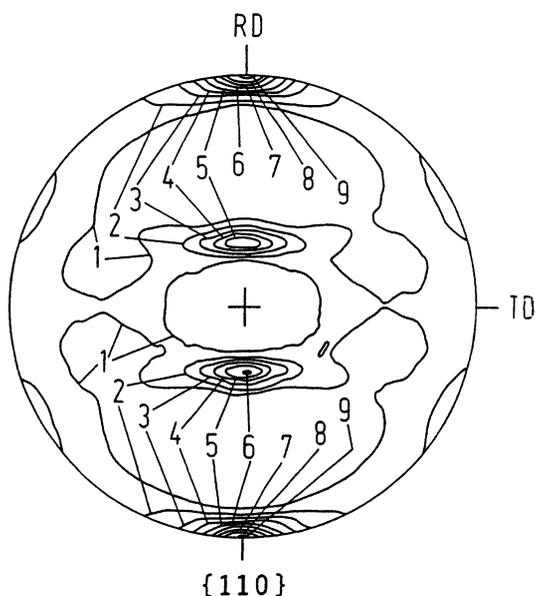
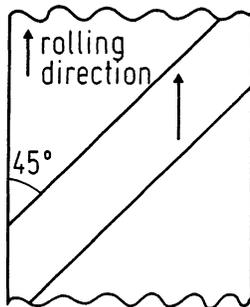


Figure 6c.  $\{110\}$  polefigure for Armco-iron cold-rolled 90%, as measured by combined Transmission/Reflection technique.

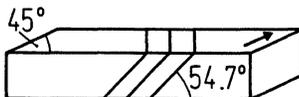
#### DEVELOPMENTS OF THE BASIC TECHNIQUE

The technique as described above is suitable for thin sheet material of less than about 4 mm thickness and where adequate supplies of material are available. This is usually the case in investigation of such properties as formability.

Where the material is thicker or in very limited supply extensions to the technique are possible but with reduced sampling or accuracy. These developments are summarised in Figures 7 and 8.



a. block is cut from rolled plate



b. slices are cut from block



c. slices are bonded edge-to-edge

Figure 7. Composite specimen technique for thick plate.

Samples for the measurement of texture in plate material are produced by milling out a slice at  $45^\circ$  to the rolling direction and of full plate thickness. Sufficient sections are cut from the slice at  $54.7^\circ$  to the rolling plane and bonded edge to edge (Figure 7), to give a composite sample of the required size. This allows only single quadrants of the polefigure to be measured but the differences between quadrants are normally small and more quadrants can be measured if required. Care must be taken when measuring the specimen that the volume of material sampled by the X-rays is representative of the full plate thickness; it is possible with, for example, only two sections bonded together that the circular area irradiated by X-rays considerably over-represents the surface of the material.

Where shortage of material is an insurmountable problem, another technique can be used. This is illustrated in Figure 8. Each individual coupon is cut into a number of pieces which are then bonded obliquely with the aid of a jig (Figure 9). The composite is held fast in the jig once the glue dries and the surface can be milled flat prior to grinding. This technique is similar to that of Leber<sup>5</sup> who has estimated the

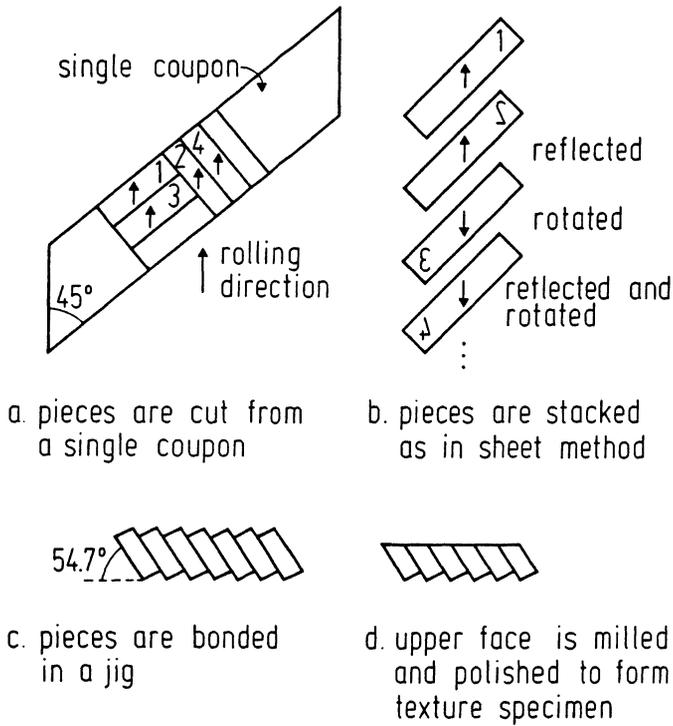


Figure 8. Composite specimen technique for restricted volume of material.

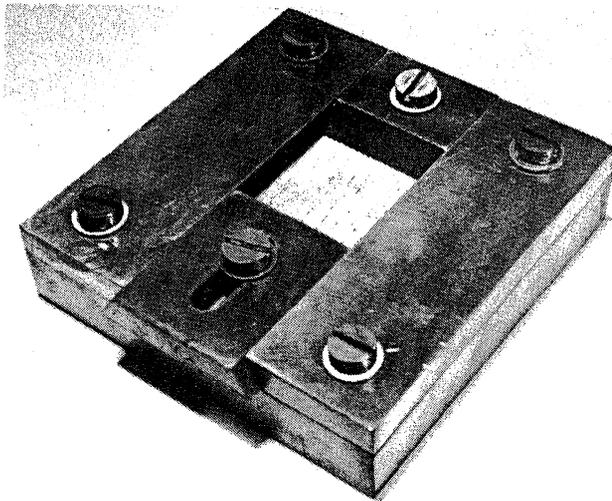


Figure 9. Photograph of jig used in procedure for small amounts of material.

accuracy as  $\pm 2^\circ$ . This is a good deal less accurate than that using the basic procedure described but the effect of the small misalignments will be to spread the texture about the correct value and lower its severity. The actual position

of the centre of the texture components is unlikely to be affected significantly.

#### ACKNOWLEDGEMENTS

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