A new procedure for relocating mineral grains for microprobe analysis

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Abstract

A new procedure is described for locating mineral grains in geological samples prepared for microprobe analysis. This procedure uses a digital image of the entire sample surface to select points of interest. These features of interest must be visible in this external image. Once the sample is mounted on the specimen stage of the microprobe, the digital image is used as a 'map' to relocate minerals for analysis. The image can be recorded using either a video camera, fitted with a macro zoom lens, or a flat-bed scanner. A calibration procedure has been developed in which two indexing points are used to calculate the instrument stage coordinates from the pixel coordinates of the digital image. Following this calibration, the stage coordinates of any feature visible in the digital image can be displayed to facilitate rapid relocation. The procedure was evaluated by relocating magnetite grains visible in an optical image of a geological thin section. The repositioning accuracy in relocating 28 magnetite grains distributed over an area of about 20 × 30 mm was found to be 157 ± 102 μm (one standard deviation) from the video camera image and 48 ± 28 μm from a flat bed scanner image. The former image was thought to suffer from some optical distortion. The procedure may be applied to any microprobe instrument fitted with a digital x–y specimen stage. Future applications in relocating features visible in autoradiographs are currently being evaluated.

KEYWORDS: microprobe analysis, mineral grains, grain location.

Introduction

OVER the last three decades, there have been considerable advances in the development of microprobe techniques (e.g. electron microprobe, scanning electron microscope, ion probe, laser ablation techniques, etc.). However, over this period, little change has occurred in procedures used in many laboratories for examining samples to locate minerals for microprobe analysis. In geological applications, samples, normally prepared as thin sections, are examined using a petrological microscope. A trained petrologist can distinguish visually many types of silicate, carbonate and oxide minerals using transmitted light illumination (Kerr, 1959) and obtain information on opaque phases using reflected light, although unambiguous identification may be more difficult (Jambor and Vaughan, 1990). As well as obtaining qualitative mineralogical information about the sample, the user would normally select specific minerals for subsequent microprobe analysis. However, having subsequently mounted the sample in the microprobe, users may encounter practical difficulties in relocating points previously selected for microanalysis.

The first difficulty is the efficient transfer of the spatial coordinate information obtained, during the off-line examination of the sample using the optical microscope, of the mineral selected for microanalysis. Although the microprobe may be fitted with a supplementary optical microscope, the quality of the optics is rarely as good as those fitted to a petrological microscope, and the transmitted light option may not be available. Furthermore, for

Mineralogical Magazine, June 1995, Vol. 59, pp. 221–228

electron and ion probe microanalysis, specimen surfaces must be coated with carbon (or gold) causing changes in the reflectance colours compared with an uncoated sample.

A second difficulty is that the operator can easily become disorientated whilst trying to relocate the points previously identified for microprobe analysis. This problem arises because of the restricted field of view normally available to the operator once the sample has been mounted in the microprobe. It is then often difficult for the operator to orientate the current field of view in relation to the entire surface of the sample. As an illustration of this problem, the optical field of view available to the operator when using the binocular microscope to focus the specimen height of a sample in a microprobe is unlikely to exceed 100–200 μm, less than one hundredth of the total area of a typical specimen.

This problem of relocating features of interest when transferring from optical microscope to microprobe is, perhaps, one of the neglected areas of modern microprobe technology. To overcome these difficulties, several procedures have been used in the past. One is to mark the surface of thin sections by circling features of interest using a diamond-tipped or ink pen, each circle being linked by a tie-line indicating the route to be taken around the sample. Alternatively, photomicrographs at the appropriate magnification can be recorded and used to assist in the relocation of features of interest. At a more sophisticated level, some laboratories have developed digitizing *x-y* stages fitted to an off-line optical microscope, the movement of which replicates that of the microprobe specimen stage. The coordinates of features identified during a preliminary off-line examination can then be recorded and read directly into the microprobe instrument. To work effectively, the *x-y* stage must be purpose-designed to replicate the dimensions of the microprobe stage (M.T. Styles, D.J. Bland and R.K. Walker, British Geological Survey, pers. comm.). A number of image analysis techniques have been developed to facilitate the automatic relocation of minerals. Most of these procedures involve the analysis of backscattered electron images using additional X-ray data where appropriate. Individual images have, of necessity, a limited field of view but these procedures may be adapted by recording and analysing a series of scanned images, in sequence, over the surface of the sample. Specialized instrument-control, acquisition and analysis systems are required to undertake these measurements, which take up a relatively large amount of instrument time. However, such procedures can be very successful in locating and quantifying rare mineralization in geological samples (Jones and Gavrilovic, 1968; Harrowfield *et al.*, 1988; Mainwaring and Petruk, 1989; Petruk, 1989; Walker *et al.*, 1989; Walker and Jones, 1990). At least one system, based on integrating an image analysis system, scanning electron microscope and energy dispersive x-ray spectrometer, can accept direct image data from rock slabs and photographic prints as a basis for quantifying the mineralogy of samples (Walker and LeCheminant, 1989).

In this paper, we describe an alternative approach to this problem of relocating features of interest for microprobe analysis. The technique is based on recording a digital image of the entire surface of the specimen. This image is then used as a 'map' which is available both to record the results of a preliminary off-line examination of the specimen on an optical microscope and then to relocate features for microanalysis once the sample has been mounted in the microprobe instrument. As developed in this work, digital images are stored and manipulated specifically in an Apple Macintosh™ computer environment. However, the development is not dedicated to any particular type of microanalysis instrument. Indeed, the only real restriction is that the microanalysis instrument (whether it is an electron microscope, scanning electron microscope or even an optical microscope) must be fitted with an *x-y* sample stage with digital readout and a resolution in movement appropriate to the application. In this paper, the principles and performance of the procedure are discussed and the limitations on repositioning accuracy presented by the analysis of a test sample.

Experimental procedure

Specification of instrumentation

Instrumentation used in this work was as follows:

Video camera. Sony EVI-1011P high resolution video camera fitted with a Computar 10 × macro zoom lens (752 × 582 pixels).

Scanner. Canon CLC-10 400 dpi flat bed scanner with enhancement of images (if necessary) using the proprietery software: 'Photoshop™' (Adobe Ltd.).

Microcomputer. Apple Macintosh™ IIfx computer (8 Mb memory) fitted with a 40 Mb internal hard disc and a Screen Machine™ 690 × 450 pixels image capture board and a 240 Mb external hard disc.

Summary of procedure

The sequence of operations required for the relocation of features of interest using instrumentation shown schematically in Fig. 1 is as follows:

(1) Record a digital image of the surface of the sample using either a video camera fitted with a macro zoom lens or a flat bed scanner.

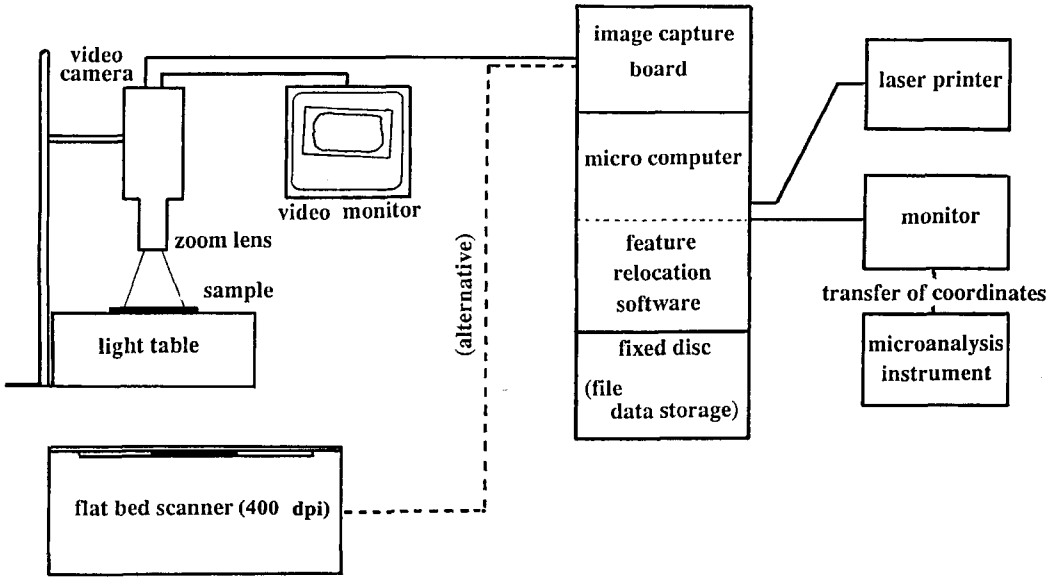


FIG. 1. Schematic diagram showing the instrumentation used to record digital images from a video camera or flat bed scanner for the relocation of minerals of interest.

(2) Place the sample on the stage of the microscope/microprobe instrument and use the procedure described in this work, based on the selection of two indexing points, to calibrate the coordinates of any pixel in the digital image in terms of the stage coordinates of the specimen as mounted for analysis on the specimen stage of a microscope/microprobe.
(3) Move the electronic pointer displayed on the digital image to highlight any feature of interest that is visible in the image. By use of a continuous display of the corresponding stage coordinates of the pointer, any highlighted feature can be relocated rapidly by driving the specimen stage to the indicated position.
(4) Minor adjustment can then be made to the microscope/microprobe stage to centre the feature of interest on the cross-wires so that the appropriate analysis can be undertaken.

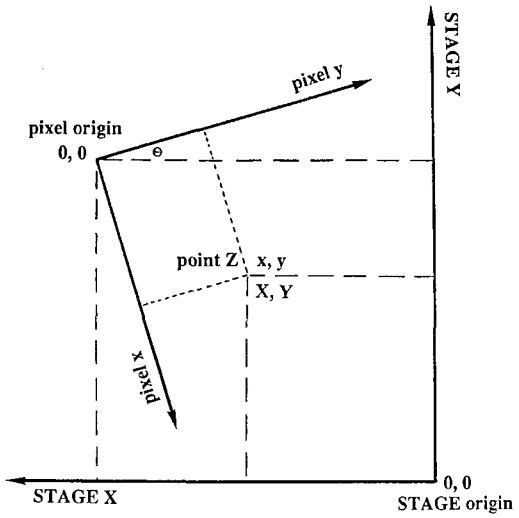
Coordinate transformation algorithm

The algorithm that recalculates pixel coordinates as stage coordinates is based on a trigonometric expression which converts the coordinates of a point Z in the digital image which has pixel coordinates (x, y) into the stage coordinates (X, Y) of this feature visible on the sample, now mounted on the microanalysis instrument specimen stage (Fig. 2). The model allows for a rotation (θ) between the two coordinate sets. However, it is assumed that the magnification conversion factor is the same for both x and y axes (i.e. that there is no difference in magnification between these axes) and that both coordinate arrays are orthogonal. Thus, the model used in this work does not account for any distortion in the digital image, a limitation that is discussed further below, nor any non-linearities in the scaling of the stage coordinates.

During the development of the transformation algorithm, it became apparent that the form of the trigonometric function varied according to the relative orientation of the stage coordinates. Whereas the digital image always had a coordinate origin in the top left hand corner, the orientation of axes of a specimen stage varied according to instrument design and indeed could have one of four orientations (Fig. 3). When initiating the program used in this work, the operator must specify which orientation is appropriate for the instrumentation in use to select the correct transformation function. As an illustration, the general formula for transforming pixel coordinates (x, y) into stage coordinates (X,Y), assuming the stage is orientated as in arrangement (a) in Fig. 3 is as follows:

$$X = X_0 - F[x/\cos\theta + (y - x\tan\theta)\sin\theta]$$

$$Y = Y_0 - F[(y - x\tan\theta)\cos\theta]$$



Calibration of pixel coordinates to stage coordinates

FIG. 2. Diagram showing the inter-relationship between the digital image array (x, y) and the stage coordinate array (X, Y) used as the basis for developing the trigonometric relationships between the image and stage coordinates of a feature of interest (Z). It is assumed that the two coordinate arrays are rotated through an angle θ relative to each other. Other symbols are identified in the text.

where F is the linear magnification factor between the pixel and stage coordinate arrays (i.e. μm per pixel), X_0 and Y_0 are the offsets between the origins of these coordinate arrays and θ is the angle of rotation between the two arrays. All these factors (F, X_0, Y_0, θ) are quantified during the calibration procedure (see next section).

Calibration procedure

The calibration procedure is based on correlating the X, Y stage coordinates of two indexing features located on the sample mounted in the microprobe with the position of these features visible in the digital image. An electronic pointer is used to highlight each of these features in the digital image in turn and the corresponding stage X, Y coordinates entered manually. The calibration software can then calculate an exact solution for all the unknowns in the transformation function, namely, the linear magnification factor (F), the angle of rotation (θ) and the offsets (X_0, Y_0)between the origins of the x and y axis pixel and stage coordinate arrays (see above). Stage coordinates of any feature designated by the electronic pointer on the digital image can then be displayed.

Since the calibration procedure uses differences in the coordinates of the indexing features along both x and y axes, uncertainties can be minimized by selecting features which are near the diagonal corners of a sample. Suitable features for calibration include the centres of small opaque minerals or distinctive angular intersections in the grain boundaries of larger mineral phases. If the sample is featureless, opaque graphical symbols (e.g. 'Letraset™') may be applied to the sample. Experience indicates that if such symbols are used, an intersection at right angles (as in the letter 'L') can be located more easily than an acute intersection (as in the base of the letter 'V'), since when viewed at high magnification, it is then easier to compensate for small irregularities when predicting the point of intersection. Furthermore, each symbol used should be distinctive and assymetric to avoid orientation difficulties when examining the sample at high magnification using a microscope with lateral inversion.

Resolution limitations

When comparing the resolution (μm per pixel) in digital images of a sample recorded with either a video camera or a flat bed scanner, account must be taken of the size of the pixel array available in relation to the size of the recorded image. In the case of the video camera used in this work, the size of the image array was restricted by the capacity of the digitizing board (690 × 450 pixels). By changing the magnification of the zoom lens, the image of a thin section could be adjusted to fill this pixel array. In contrast, the flat bed scanner recorded images at fixed magnification, dictated by the resolution of the scanner (400 dots per inch for the instrument used in this work). Thus, calculations show that a 30 × 20 mm^2 thin section would be recorded as a 472 × 315 pixel image.

In both cases, the digitizing process places a basic limitation on the spatial resolution with which any feature can be relocated, based on the separation of adjacent pixels. Thus, assuming the specimen has an area of 30 × 20 mm^2, the corresponding pixel-to-pixel resolution of the video camera image was about 44 μm and that of the flat bed scanner was about 64 μm (these measurements representing rectilinear distances along the x or y axes). These image resolution parameters are much larger than the spatial resolution of specimen stages fitted to microprobe instrumentation which is typically less than a micrometre.

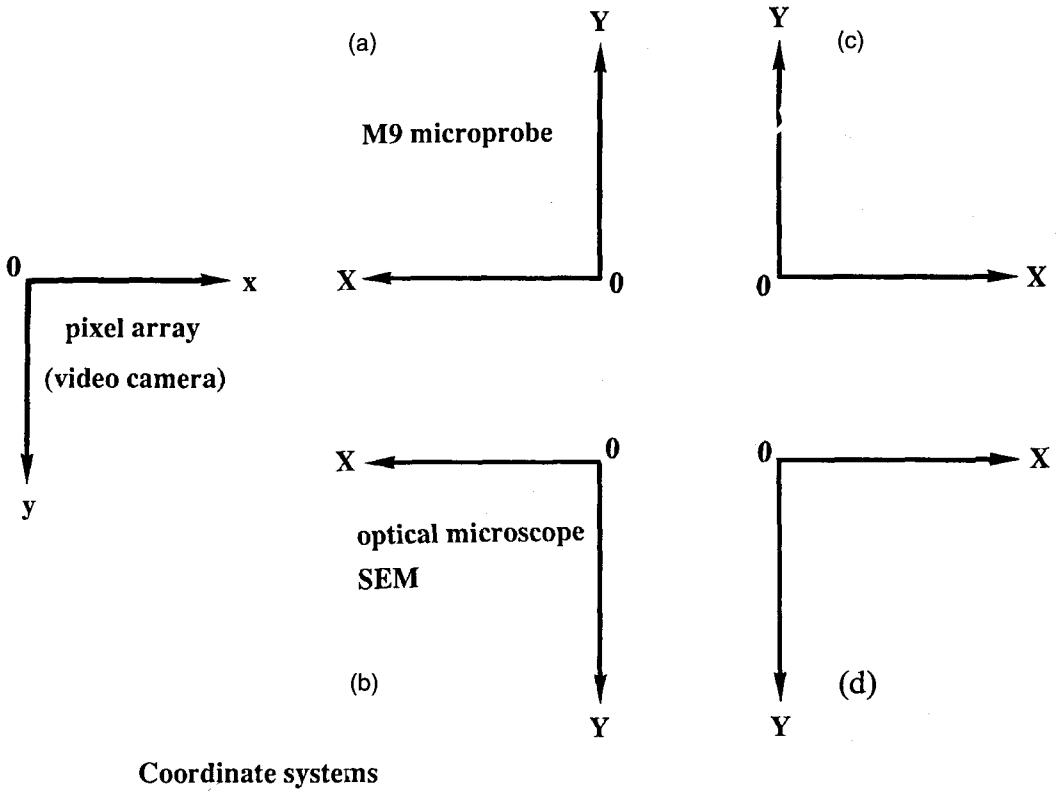


FIG. 3. Orientation of the four possible configurations of the stage coordinate array (X, Y) in relation to the digital image array (x, y). The stage arrays that correspond to three instruments available at the Open University are marked.

Operating specification

Having carried out the calibration procedure described above, the operator may use the mouse to move the electronic pointer to designate any feature visible on the digital image and obtain a continuous display of the corresponding stage coordinates. This relocation procedure can never be accurate down to the micrometre level owing to limitations in pixel-to-pixel resolution discussed above, as well as distortions that may be introduced into the image during acquisition and possibly mechanical discrepancies in the movement of the specimen stage. Having driven to the predicted stage coordinates, the operator must make final adjustments under manual control to centralize the feature of interest. Taking account of the unavoidable relocation errors referred to above, an appropriate objective for this procedure is that after moving the specimen stage to the predicted position of any feature of interest, the actual position should always lie within the current field of view, and that this specification should be maintained over the entire surface of the sample. In terms of the field of view of typical microprobe instrumentation, the maximum acceptable repositioning error is about 200 μm over an area of 20 × 30 mm^2.

The system developed in this work requires the manual transfer of stage coordinates predicted by the operating program to the microprobe. This procedure allows complete flexibility in applying the procedure to any microprobe or microscope instrumentation fitted with a digitised x-y specimen stage. However, if dedicated to a particular instrument, it would be a relatively simple task to interface the software to allow electronic transfer of coordinate data.

Results and discussion

Evaluation of relocation accuracy

To evaluate the repositioning accuracy, the proposed procedure was applied to a thin section prepared from

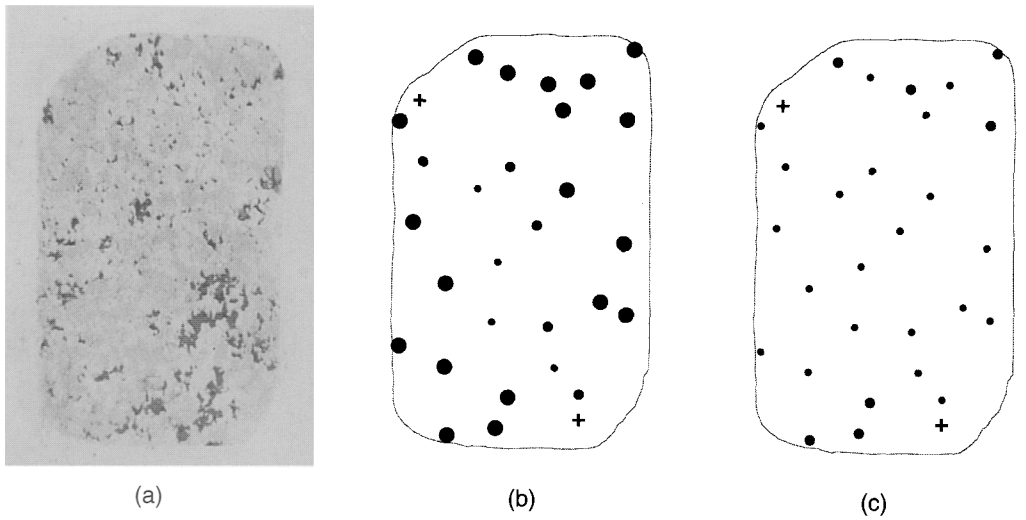


FIG. 4. Digital image (*a*) of the orthopyroxene-bearing granodiorite test sample showing, small black crystals of magnetite with minor amounts of biotite. Repositioning errors observed when using digital images recorded by video camera (pixel-to-pixel resolution = 46 μm) and flat bed scanner (pixel-to-pixel resolution = 63 μm) are shown in (*b*) and (*c*) respectively. Circles increase in size according to the following scale: Small circles — repositioning error < 1 pixel, intermediate circles — repositioning error 1 to 2 pixels, large circles — repositioning error > 2 pixels. Crosses mark the two mineral grains used in the calibration procedure.

a sample of orthopyroxene-bearing granodiorite from the Prince Charles Mountains, Antarctica. This specimen was chosen because it contained a large number of approximately circular magnetite grains (and some biotite) in the 100 to 300 μm size range which were readily visible in a digitized image and easy to relocate on the original thin section (Fig. 4*a*). Two minerals at diagonal corners of the sample were selected to calibrate the image (see Fig. 4*b,c*) and 28 individual mineral grains, selected to cover the entire area of the sample were relocated on the specimen mounted in the *x-y* stage fitted to an optical microscope, using coordinate data predicted from the digital image. Repositioning errors were recorded as the difference in μm between the predicted and actual position of the centre of the selected mineral grain. This experiment was carried out using two digital images. One image was recorded with the video camera/macro lens assembly at a pixel-to-pixel resolution of 46 μm. The other was recorded using the 400 dpi flat bed scanner, at a pixel-to-pixel resolution of 63 μm. Results are listed in Table 1 and displayed graphically in Fig. 4. These data indicated that the mean relocation discrepancy based on the video camera image was 157 ± 102 μm and that from the scanned image was 48 ± 28 μm, a result which is surprising in view of the less favourable pixel-to-pixel resolution of the image recorded on the flat-bed scanner. Examination of the spatial distribution of mineral grains for which relocation discrepancies were largest (Fig. 4) indicates that the video camera image appears to suffer from some distortion effect, since the largest discrepancies are found at the edges of the image. In both cases, the mean relocation discrepancy was within the target figure of ±200 μm but a number of values derived from the video camera image exceeded this figure. These results indicate that the scanner is the preferred form of image capture when maximum repositioning accuracy is being sought, because of the higher degree of linearity in the digitization process.

Although the developments described here have been evaluated from a simple optical image of the sample, work is now in progress to extend this application to autoradiography images of a sample that contain information about the spatial distribution of selected mineral phases. It is expected that the full benefits of the procedure will be exploited in applications such as beta autoradiography (Potts, 1984), fission track autoradiography (e.g. Wollenberg, 1973), alpha autoradiography (e.g. Basham and Easterbrook, 1977) and X-ray excited optical luminescence autoradiography (Potts and Tindle, 1990).

TABLE 1. Repositioning error (μm) on 28 grains selected over the surface of the orthopyroxene-bearing granodiorite test sample (see Fig. 4 for positions)

Video camera image (pixel-to-pixel resolution = 46 μm)						
65	23	28	36	254	306	232
100	186	247	170	226	135	136
75	161	187	337	143	83	62
264	72	140	204	418	95	14
mean = 157.1 μm; standard deviation = 102.1 μm						
Flat bed scanner image (pixel-to-pixel resolution = 63 μm)						
41	36	30	67	36	61	93
71	79	93	34	58	48	16
46	79	59	79	11	13	3
29	11	22	27	102	56	31
mean = 47.5 μm; standard deviation = 27.8 μm						

Acknowledgements

The authors are very grateful to John Sheraton (Australian Geological Survey, Canberra) for providing the test sample, to the Open University Research Committee for the award of a grant to purchase instrumentation and to the reviewers for suggesting improvements to the original manuscript.

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[*Revised manuscript received 24 August 1994*]