carried out by the program is knowledge of the particle orientation in the image. This guarantees that the straight lines fitted to the particle curve are parallel to the {111} facets. Even though the program works for any particle orientation in the image the result of the measurement depends strongly and in a non-linear fashion on the particle orientation. In order to obtain comparable results all particles were scanned with the c-axis aligned vertically in the image. After selecting the c-axis to be vertical the error due to misalignment from this position was determined. From the image output files it can be seen that a misalignment of 1° is hardly detectable. At a misorientation of 2° the discrepancy between the actual {111} facets and straight lines fitted to these facets becomes obvious, see Fig 4. The error averaged over ±1° misalignment amounts to 0.44% for c/a and 0.56% for the contact angle θ with respect to values obtained for no misalignment.

IMAGE ACQUISITION

Specimen tilt

During image acquisition in the TEM the alignment of the particle with respect to the <110> zone axis determines the projected particle shape. Only when the particle is oriented parallel to a <110> zone axis are two sets of {111} facets precisely on edge. A tilt out of the exact zone axis orientation will lead to a distorted projection of the shape that results in falsified contact angles and distances. To estimate this error the specimen was deliberately tilted out of the <110> zone axis in the microscope along the <020> and <111> Kikuchi bands by 3° and 1° respectively. Images taken in these orientations were evaluated together with an image of the same particle oriented parallel to the <110> zone axis. The maximum error determined from a comparison of the results amounts to 1.32% for c/a (along <020>) and 0.97% for the contact angle θ (along <111>).

Image exposition and contrast levels

Over and under exposure as well as reduced contrast levels will affect the accuracy with which the binary image approximates the true particle shape and therefore influence the result of the measurement. For example it can be seen in Fig 1a how the intensity at the spherical caps of the particle is lower and fades gradually compared to the intensity at {111} facets that ends in a sharp line. A high intensity (overexposure) at the spherical caps will give a smaller radius of curvature in the binary image than a low intensity (underexposure, flattened spherical caps). Similarly a variation in contrast value affects the intensities at the particle edges. We assume the shape obtained from an image recorded in auto-exposure and with contrast levels adjusted automatically to be the true particle shape and compare it to images recorded in over and under exposure or with deliberately misadjusted contrast levels. The errors estimated for variation in exposure time is highest at underexposure and amounts to 0.55% for c/a and 0.84% for the contact angle θ. Variations in contrast had a smaller effect with a maximum error of 0.28% for c/a and 0.24% for θ.

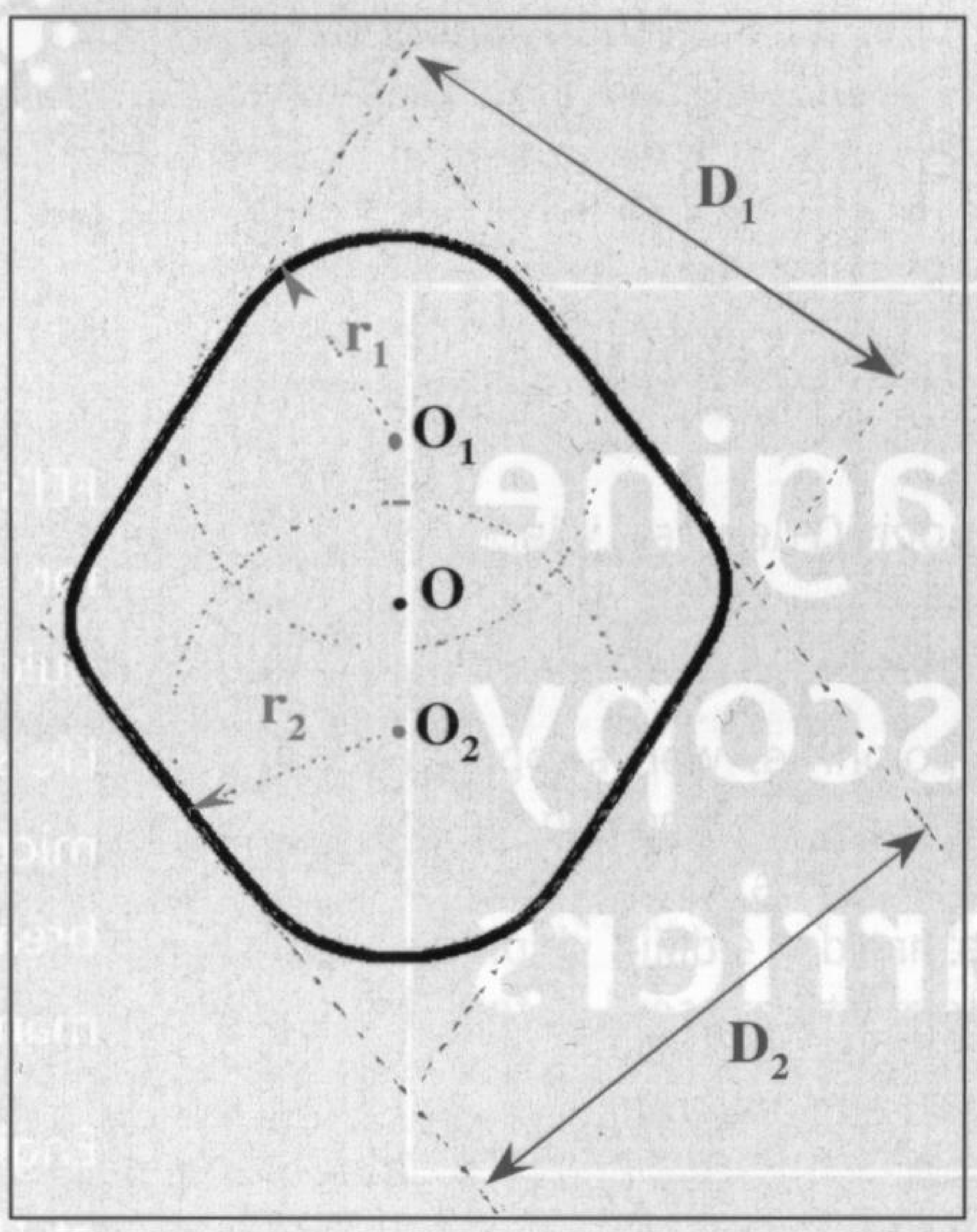

Figure 2:
Parameters measured to compute contact angle θ and aspect ratio c/a. Distances D1 and D2 between the {111} facets, the radii r_1, r_2 of curvature of the spherical end caps, the coordinates of the circle centers O_1 and O_2 of the spherical caps and the coordinates of the center of gravity of the particle O.

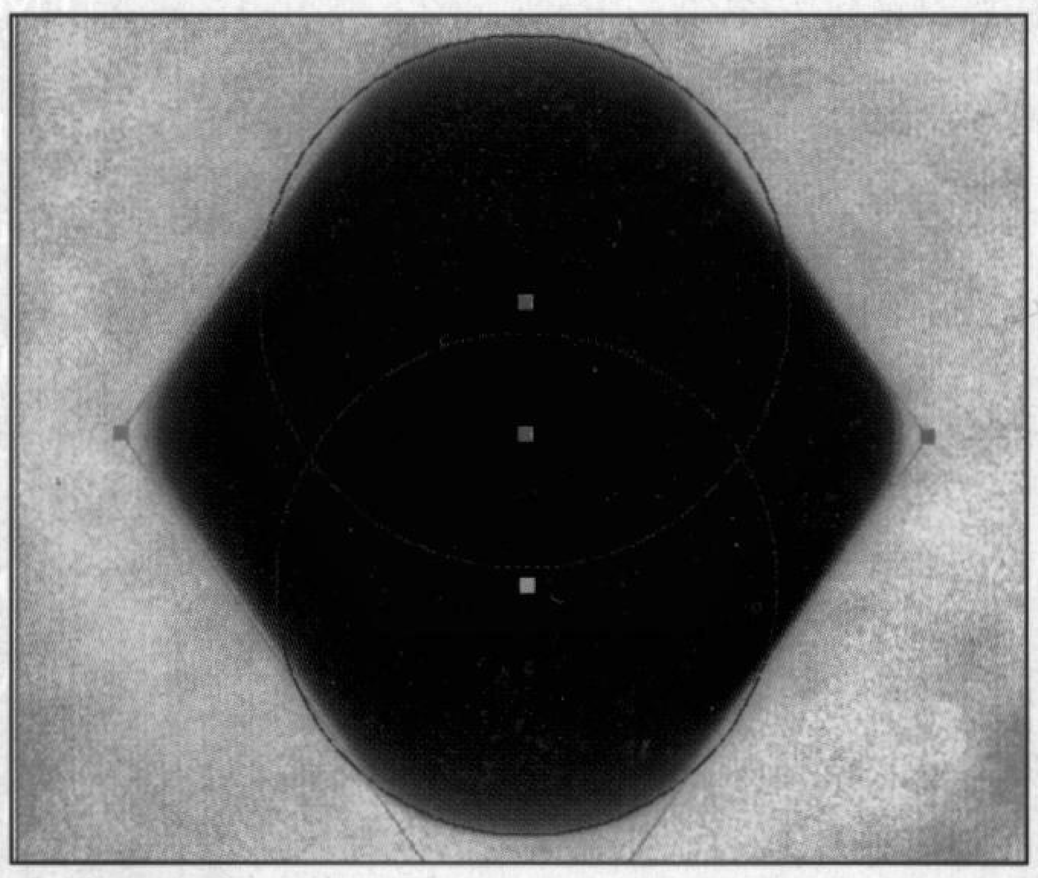

Figure 3:
Image outputfile with curve of particle outline, straight lines fitted to {111} facets and circles fitted to spherical end caps.

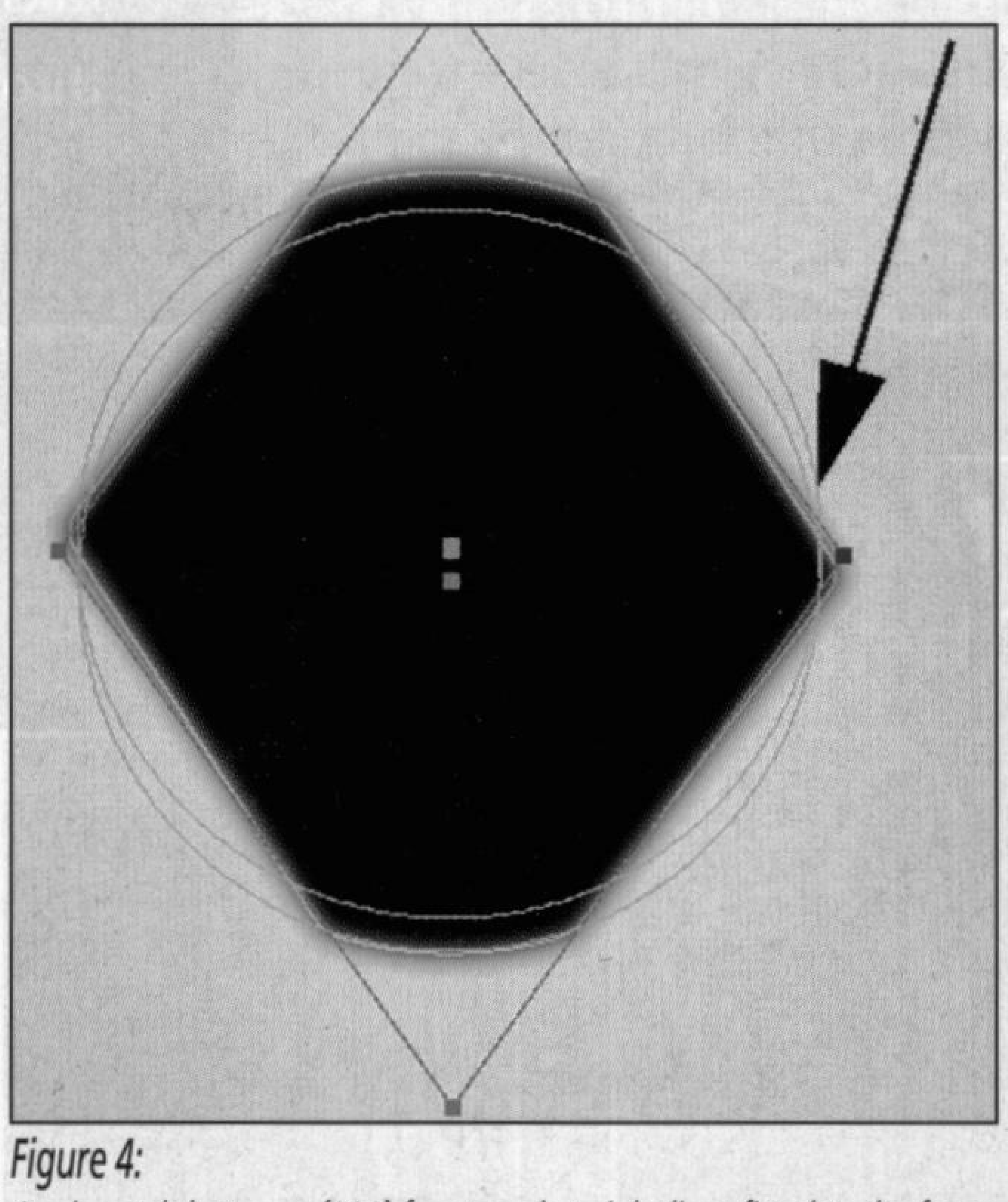

Figure 4:
A mismatch between {111} facets and straight lines fitted to the facets is clearly visible (arrow). It results from a 2° misalignment between particle axis and the program reference system.

CONCLUSION

We developed and tested a method to measure particle shapes accurately and reproducibly. The errors inherent in the program are the limitation of accuracy due to the number of data points available for evaluation and the dependence of the results on the orientation of the object in the image. The errors caused by both variables can be kept below 1% by choosing an image resolution corresponding to 320 pixels on the particle diameter and by keeping the particle orientation constant in all images. The effect of experimental errors such as particle misalignment in the TEM, intensity variations around the particle outline and variation of image contrast was estimated using the evaluation program. It was found that the deviation from the exact orientation in the <110> zone axis has the strongest impact on the results. A continuous control of the particle orientation throughout the experiment is therefore necessary. The effect of intensity variation at the spherical {020} caps is stronger for underexposure than overexposure while variations of the image contrast have a negligible contribution.

The application of the program to monitor the shape changes of small liquid Pb particles in solid Al at different temperatures during a heating and cooling cycle enabled us to resolve the controversy about the anisotropy of the interfacial energy in the system Pb-Al [5]. From the results of a large number of measurements we established the hysteresis in shape between heating and cooling and obtained the true equilibrium shape. The concept of this new method, i.e. to treat the particle outline as a mathematical curve and to exploit the particle geometry to derive a measure for the shape, has the potential to be adapted to similar problems concerning the measurement of shape and physical properties.

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