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TITLE:

Quantitative Determination of Aggregate Surface Morphology at the Interfacial Transition Zone (ITZ)

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KEYWORDS:

Interfacial transition zone (ITZ), aggregate surface morphology, model concrete, SEM-BSE, digital image processing method, K-means clustering

SUMMARY:

Hereby, we proposed a protocol to illustrate the effect of aggregate surface morphology on the ITZ microstructure. The SEM-BSE image were quantitatively analyzed to obtain ITZ's porosity gradient via digital image processing and a K-means clustering algorithm was further employed to establish a relationship between porosity gradient and surface roughness.

ABSTRACT:

Here, we present a comprehensive method to illustrate the uneven distribution of the interfacial transition zone (ITZ) around the aggregate and the effect of aggregate surface morphology on the formation of ITZ. First, a model concrete sample is prepared with a spherical ceramic particle in roughly the central part of the cement matrix, acting as a coarse aggregate used in common concrete/mortar. After curing until the designed age, the sample is scanned by X-ray computed tomography to determine the relative location of the ceramic particle inside the cement matrix. Three locations of the ITZ are chosen: above the aggregate, on the side of the aggregate, and below the aggregate. After a series of treatments, the samples are scanned with a SEM-BSE detector. The resultant images were further processed using a digital image processing method (DIP) to obtain quantitative characteristics of the ITZ. The surface morphology is characterized at the pixel level based on the digital image. Thereafter, *K*-means clustering method is used to illustrate the effect of surface roughness on ITZ formation.

INTRODUCTION:

At the mesoscopic scale, cement-based materials can be regarded as a three-phase composite comprised of the cement paste, the aggregate, and the interfacial transition zone (ITZ) between them[1-2]. The ITZ is often treated as a weak link since its increased porosity could act as channels for the ingress of aggressive species[3-4] or provide easier pathways for crack growth[5-11]. Subsequently, it is of great interest to precisely characterize the properties of the ITZ to evaluate and predict the macro performance of the cement-based materials.

To investigate the ITZ, there has been excessive research on its microstructural features, forming mechanisms, and influencing factors[12-14] using both experimental and numerical methods. Various techniques have been coupled for ITZ characterization including: mechanical tests, transport tests, mercury intrusion porosimetry (MIP) tests[15-16] and nano-indentation[17]. It is widely accepted that the ITZ is mainly caused by the wall effect, as well as water film, micro-bleeding, one side growth, and gel syneresis[18].

With the development of digital image processing method (DIP) in the last two decades[19], the morphological characteristics of the ITZ (e.g., volume fraction, thickness, and porosity gradient) can be quantitatively determined. Based on examination of the plane sections using scanning electron microscopy (SEM) with a backscattered electron detector (BSE), the three dimensional (3D) features of ITZ can be derived from the 2D results via stereology theory[20]. Like the SEM-BSE technique, the nano-indentation technique is also based on the examination of polished surfaces, but it more focuses on the elastic modulus of the existing phases[21]. However, in both SEM-BSE analysis and the nano-indentation test, the ITZ thickness may be overestimated as the examined cross section rarely goes through the normal direction from an aggregate surface[22]. However, coupling this with fluorescent 3D confocal microscopy, the overestimation of ITZ could be eliminated and a real ITZ porosity and anhydrous cement content could be obtained[23].

Previous studies of influencing factors mainly focused on the cement paste, ignoring the role of the aggregate and its surface texture[24-26]. Since the shape and morphological properties of the aggregate have been extensively described based on quantitative analysis of digital slices obtained from SEM or X-ray computed tomography (X-CT)[27-28]. However, no research focusing on the effect of the aggregate surface texture on the formation of ITZ region has been performed.

Hereby, we present a protocol to investigate the effect of aggregate surface morphology on the ITZ microstructure formation based on quantitative analysis of SEM-BSE images and a K-means clustering algorithm. A model concrete sample was prepared with spherical ceramic particle acting as the coarse aggregate. X-CT was used

to roughly determine the relative location of the particle in the opaque cement matrix before halving the sample. After processing to obtained SEM-BSE images, the uneven distribution of ITZ around single aggregate was observed. Also, an index surface roughness (SR) describing the aggregate surface texture at the pixel level was defined. The K-means clustering algorithm, originally used in the area of signal processing and now widely used for image clustering[29-30], was introduced to established a relationship between surface roughness (SR) and porosity gradient (SL).

PROTOCOL:

1. Preparation of the model concrete with a single ceramic particle

1.1. Mold preparation

1.1.1. Use a brush to clean the mold (25 mm x 25 mm x 25 mm) and ensure that the inner surfaces of the mold are impurity-free.

1.1.2. Use another brush to uniformly apply diesel oil on the inner surfaces of the mold for easier mold-release.

NOTE: Here, we did not use the common mold for mortar or concrete preparation. As the ceramic particle is around 15 mm in diameter, a cubic plastic mold around 30 mm in length is used for sample preparation. Ensure that the size of plastic mold is larger than the ceramic particle.

1.2. Molding the model concrete

1.2.1. Weigh 1,000 g of cement and 350 g of water with an electronic balance (the designed water to cement mass ratio is 0.35).

1.2.2. Wipe the 5 L mixing pot with a wet towel to moisten it. Add 350 g of water and 1,000 g of cement into the mixing pot sequentially. Place the mixing pot on the mixer and raise it to the stirring position.

1.2.3. Mix at 65 rpm for 90 s, and let the mixture stand still for 30 s. During this period, scrape off the paste on the inner wall of the pot. Then, mix at 130 rpm for another 60 s.

1.2.4. Remove the pot from the mixer and put the ceramic particle into the paste, manually mixing it with the cement paste thoroughly.

1.2.5. Half fill the mold with well mixed fresh cement paste.

1.2.6. Place the ceramic particle on the top surface of the cement paste and fill the rest of the mold with cement paste. Wipe off excess cement paste with a scraper knife and vibrate the mold on a vibrating table for 1 min at 50 ± 3 Hz.

1.2.7. Seal the mold surface with cling film to prevent moisture evaporation.

1.3. Curing

1.3.1. Cure the specimen in a curing room for 24 h (20 ± 1 °C and 95% ± 5% relative humidity).

1.3.2. Remove the specimen from the mold and further cure the specimen for 28 d under the same environmental conditions.

2. Scanning electron microscopy preparation

2.1. Determination of the ceramic particle inside the matrix

2.1.1. Scan the specimen with X-ray computed tomography to obtain a stack of slices[31].

2.1.2. Roughly choose the slice where the ceramic particle appears to be largest. Fit the boundary of the ceramic particle with a circle and determine the center of the circle as the geometrical center of the ceramic particle. Due to the gray value difference between cement matrix and ceramic particle, a rough boundary of the particle appears on each CT slice (**Figure 1**).

2.2. Cutting

2.2.1. Cut the cubic specimen into two parts through the geometrical center of the ceramic particle in a cutting machine. **Figure 1**[32] is a schematic map showing the cutting direction.

NOTE: The ceramic particle was split into two equal parts, while the specimen was not cut into two exactly equal halves. If the ceramic particle is in the exact center of the cubic specimen, the specimen will be split into two equal halves. However, in a real situation, the ceramic particle typically is not in the exact center of the specimen.

2.3. Hydration termination

2.3.1. Immerse the two parts into isopropyl alcohol (≥99.5%) for 3 days at room temperature to remove the unbounded water and terminate the internal hydration process. Replace the isopropyl alcohol solution every 24 h.

2.3.2. Place the two parts in the vacuum drying even for 7 days to dry the sample at temperature of 40 °C.

2.4. Solidifying the microstructure

2.4.1. Use a finger to smear the inner surface of two cylindrical plastic molds (31 mm in diameter and 25 mm in height) with demolding paste. The molds are all bottom removable.

2.4.2. Place each piece of the sample into each mold with the surface to be examined facing downward.

2.4.3. In a paper cup, weigh 50 g of low viscosity epoxy resin and add another 5 g of hardener. Manually stir the mixture with a wooden stick for 2 min.

2.4.4. Put the mold into the cold mounting machine along with the paper cup with the mixture.

2.4.5. Start the vacuum on the cold mounting machine and pour the epoxy resin into the mold until it merges with each sample.

2.4.6. Keep the mold in the cold mounting machine for 24 h until the epoxy resin hardens.

2.4.7. Remove the bottom of each mold and squeeze out the sample. Store the sample in a vacuum drying oven.

2.5. Grinding and polishing

2.5.1. Grind the sample with SiC paper and alcohol as a lubricant on an automatic polishing machine at the speed of 300 rpm in the following sequence for 3 min each: 180 grit, 300 grit, 600 grit, and 1200 grit.

2.5.2. Attach the flannelette to the turntable of the automated polishing machine.

2.5.3. Polish the sample on the flannelette with diamond paste of 3 µm, 1 µm, and 0.25 µm for 15 min at the speed of 150 rpm, each.

2.5.4. Remove the debris in an ultrasonic cleaner with alcohol as the cleaning solvent after each grinding and polishing step.

2.5.5. Store each sample in a plastic box of similar size to the sample with each surface to be examined facing up to avoid cause any scratches on the testing surface.

2.5.6. Keep the boxes containing the samples in a vacuum dry oven[32].

NOTE: The grinding and polishing process could be completed on an automated polishing machine and at most 6 samples could be polished at the same time. The grinding and polishing time should be carefully chosen to obtain an extremely smooth surface for the SEM without creating height differences between the cement paste and the aggregate. A typical sample is shown in **Figure 2**[32].

3. Backscattered image acquisition and processing

3.1. Acquisition

3.1.1. Spray a thin layer of gold foil on the surface to be examined in a vacuum environment with an automatic sputter coater.

3.1.2. Place a strip of adhesive tape on the side of the sample to connect the testing surface and opposite surface and place the sample on the test bench with the testing surface facing upward.

3.1.3. Move the sample to focus the lens on region 1 as labelled in **Figure 2**[32].

3.1.4. Vacuum the SEM and change to backscattered electron mode. Set the magnification at 1,000x and carefully adjust the brightness and contrast before capturing images.

3.1.5. Move the lens along the direction of the aggregate boundary to another position of the aggregate and take another image. Repeat this moving and imaging process at least 15 times so that enough images can be obtained for statistical analysis.

3.1.6. Move the lens to region 2 and region 3 and repeat the imaging process.

NOTE: Each image should include three phases: the matrix, the aggregate, and the ITZ. Since ITZ is a narrow section existing between another two phases and hard to be distinguished, each image should include both the cement matrix and the aggregate.

3.2. Processing

3.2.1. Pre-treat the image with a best fit and 3 x 3 median filter three times to reduce the noise and enhance the boundary of different phases on ImageJ.

3.2.2. Manually capture the boundary of the ceramic particle and cut off this part from the original image using ImageJ.

3.2.3. Roughly determine the upper threshold value of pore phases by setting different threshold values and segmenting the image to compare with the original one.

3.2.4. Obtain the gray-scale distribution of the remaining part of the image. Choose two approximatively linear parts of the distribution curve just around the roughly determined upper threshold value of pore phases. Fit these two parts with linear curve and the intersection point will be set as the exact upper threshold value of this image (see **Figure 3c**[32]).

3.2.5. Use this value to do the segmentation and compare the binary image with original gray-scale image for final threshold value determination.

3.2.6. Convert the gray-scale image to a binary image with white (gray value = 255) representing pore phase and black (gray value = 0) representing solid phases.

NOTE: The exact determination of threshold value is called the overflow point method[33] since the brightness and contrast are kept the same for different images obtained from same sample. Once the upper threshold value is precisely determined, this value could be applied to other images obtained from the same sample.

4. Data processing

4.1. ITZ thickness determination

4.1.1. Delineate twenty 20 successive strips that are 5 µm in width (use the included **strip_delineation.m** file), along the captured boundary in the direction of starting from aggregate surface and going into the bulk paste (see **Figure 3d**[32]).

4.1.2. Count the number of pixels with a gray value lower than the threshold in each strip and normalize the values by the number of total voxels contained in each strip. Each normalized value will be viewed as the porosity of each strip.

4.1.3. Repeat the counting and normalization process for all the images. Average the porosity profiles of the same strip number from different images.

4.1.4. Draw the porosity distribution graph as a function of distance away from aggregate surface. Determine the inflection point on the curve where porosity become stable as the thickness of the ITZ.

NOTE: The number of strips and width of each strip could vary; make sure that the total width of the delineated strips includes all of the ITZ. According to previous research, the ITZ thickness ranges between 20-50 μm[13]. Even in some model concrete samples with an enlarged ITZ, this value does not exceed 70 μm[34-35].

4.2. Aggregate surface roughness (SR) characterization

4.2.1. Save the manually captured boundary as a curve. Fit the irregular boundary with both straight line and circle arc according to the Eq. (1) and Eq. (2) based on least square algorithm.

$$(x-a)^2+(y-b)^2=R^2 \quad (1)$$

$$Ax+By+C=0 \quad (2)$$

with (a,b) being the center of the fitting circle.

4.2.2. Define the deviations between original irregular boundary and fitting smooth curve as the surface roughness (SR).

4.2.3. For straight line, calculate the SR_s by averaging the absolute value of the perpendicular distance of the center of each pixel on the boundary to the fitting line:

$$SR_S=\frac{\sum_1^n|\frac{Ax_i+By_i+C}{A^2+B^2}|}{n} \quad (3)$$

with n being the number of pixels included in each boundary and (x_i, y_i) being the coordinates of the ith pixel on the boundary.

4.2.4. For a circle arc, define SR_C as:

$$SR_c=\frac{\sum_1^n|\sqrt{(x_i-a)^2+(y_i-b)^2}-R|}{nR} \quad (4)$$

4.2.5. Compare the value of SR_S and SR_C for each boundary and determine the minimum value as the final surface roughness for this curve (use the included **surface_roughness_calculation.m** file).

NOTE: The surface roughness of boundary should be defined against a smooth baseline curve. Both straight line and circle line were used for the following reason. Though the boundary of the spherical ceramic particle appears like a circle in 2D, some local regions appear to be more appeal to a straight line.

4.3. K-means clustering

4.3.1. Define a slope index (SL) to describe the porosity gradient within the interfacial transition zone according to the Eq. (5).

$$\mathrm{SL}=\frac{\varphi_{\max}-\varphi_{\min}}{\varphi_{\min}} \quad (5)$$

where $\varphi_{\max}$ is the value of the porosity in the first strip (0 μm to 5 μm) and $\varphi_{\min}$ is the value of the porosity in the sixth strip (25 μm to 30 μm).

4.3.2. Combine the SR and SL of each boundary to be an observation $(\mathrm{SR_i}, \mathrm{SL_i})$. And for total n boundaries and ITZs, there exist n observations to be saved as a cluster $\{(\mathrm{SR_1}, \mathrm{SL_1}), (\mathrm{SR_2}, \mathrm{SL_2}), \ldots, (\mathrm{SR_n}, \mathrm{SL_n})\}$.

4.3.3. Apply a K-means clustering[36-37] algorithm (use the included **k_means_clustering.m** file) to all the observations and subdivide them into 2 clusters: rough and smooth aggregate surface group, respectively.

4.3.4. Average the porosity distributions of ITZ in rough and smooth cluster, respectively. Compare the average porosity distribution between two clusters.

NOTE: Herein, K-means clustering is a method of vector quantization, which is originally used in signal processing and currently widely applied to cluster analysis in data mining. The aim of the method is to subdivide the observations into 2 or more subgroups.

REPRESENTATIVE RESULTS:

The porosity distribution of ITZ regions above the aggregate, on the side of the aggregate, and below the aggregate are compared and shown in **Figure 4**[32]. The porosity of the ITZ above the upper surface appears to be smaller than that on the side or above the aggregate, indicating a denser ITZ microstructure, while the ITZ below the aggregate is always the most porous due to micro-bleeding. **Figure 4**[32] shows that even around the same aggregate, the distribution is uneven.

To investigate the effect of aggregate surface morphology, the manually captured irregular boundary is fitted with straight line and circle arc, respectively as shown in **Figure 5**[32]. The blue line is the original irregular boundary, while the fitting curve is represented by the red line. For the chosen boundary it appears to be closer to a straight line.

Based on the calculations of the defined SR and SL parameters, the ITZ from varying relative locations to the aggregate surface are viewed as a whole $\{(SR_1, SL_1), (SR_2, SL_2), \ldots, (SR_n, SL_n)\}$. The K-means clustering algorithm is applied to subdivide the scattering points into two groups: a rough group and a smooth group as shown in **Figure 6**[32]. The dashed line shows that the value of SL decreases with increasing SR values.

The porosity distributions of the ITZs in the rough and smooth group are averaged and a comparison is shown in **Figure 7**[32]. At nearly every distance, the porosity of ITZ around the smooth surfaces is significantly than the porosity of ITZ around rough

surfaces, which proves that the surface morphology indeed plays an important role in ITZ formation.

Figure 1. An image of a CT slice through the equator of the ceramic sphere. This figure has been modified from[32].

Figure 2. A typical sample with highly polished surface and ready for SEM-BSE test. This figure has been modified from[32].

Figure 3. Schematic view of the BSE image analysis process: (a) original image, (b) boundary capture, (c) threshold determination, and (d) strip delineation. This figure has been modified from[32].

Figure 4. Comparison of porosity distribution of ITZ in three different relative locations to the aggregate surface. With region 1: ITZ above the aggregate (U-ITZ); region 2: ITZ on the side of the aggregate (S-ITZ); region 3: ITZ below the aggregate (L-ITZ). The error bar in the distribution curve is the standard deviation. This figure has been modified from[32].

Figure 5. Schematic map of fitting the aggregate boundary using a straight line and a circular arc: (a) original BSE image, (b) straight line fitting to the aggregate surface, (c) circular arc fitting. The image is approximately 0.19 um in width. This figure has been modified from [32].

Figure 6. Results of performing K-means clustering to partition all boundaries into K=2 clusters. This figure has been modified from[32].

Figure 7. Comparison of porosity distribution of ITZ against rough and smooth surfaces. The error bar in the distribution curve is the standard deviation. This figure has been modified from[32].

DISCUSSION:

The X-CT technique was applied to roughly determine the geometrical center of the ceramic particle to ensure that the analyzed surface is through the equator of the particle. Thus, the overestimation of the ITZ thickness caused by the 2D artifacts could be avoided[38]. Herein, the accuracy of obtained results is highly dependent on the flatness of the examined surfaces. Generally, a longer grinding and polishing time contributes to an adequately smooth surface for testing. However, due to the varying hardness between cement paste and ceramic particle, prolonged grinding and polishing time tends to create height difference between the two phases, which appears as a 100% porosity gap on the obtained BSE images. To eliminate this effect, the grinding and polishing strategy should be carefully chosen according to the properties of the materials[21, 39]. Once there exists such gap on the image, we chose to

capture the boundary along the contour of the cement paste rather the exact aggregate surface.

The ITZ properties were derived from the porosity gradient. In reality, the volume fraction of hydration products, anhydrous clinkers could also be determined from the image. By coupling using energy dispersive spectroscopy (EDS), we can obtain how the Ca/Si changes in this region, which helps to determine the characteristic parameters of the ITZ. In this paper, the ITZ thickness obtained is around 70 μm, which is larger than the value reported in previous research. Multiple factors contribute to the phenomenon. There is only one spherical ceramic particle included in such model concrete and no interactions between different aggregate particles occurs during the hydration process, which is different from the common mortars or concrete. A second factor is the insufficient mixing during the sample preparation process. The interfacial transition zone was enlarged in the concrete sample and we will further consider a better way to overcome this disadvantage.

Based on the obtained image, the irregular aggregate boundary was quantitatively described and compared at a pixel level. The K-means clustering algorithm is a powerful method for cluster analysis, which is able to subdivide observations into 2, 3, 4, or even more groups. The results of the K-means clustering are affected by the initial centroids of each cluster and, here, the Forgy method was chosen [40]. K observations were picked randomly from the n observations to serve as the original K centroids[36]. Herein, for surface roughness analysis, we also tried 3 and 4 groups. However, with increasing clusters, the porosity difference between different groups is not as distinct as dividing them into 2 groups. We will keep seeking other applications of K-means clustering method in cement and concrete research, like phase determination in nano-indentation.

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DISCLOSURES:

We hereby confirm that this manuscript is our original work and all the authors listed have approved the manuscript and have no interest conflicts on this paper.

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581 initialization methods for the k-means clustering algorithm. *Expert Systems with*
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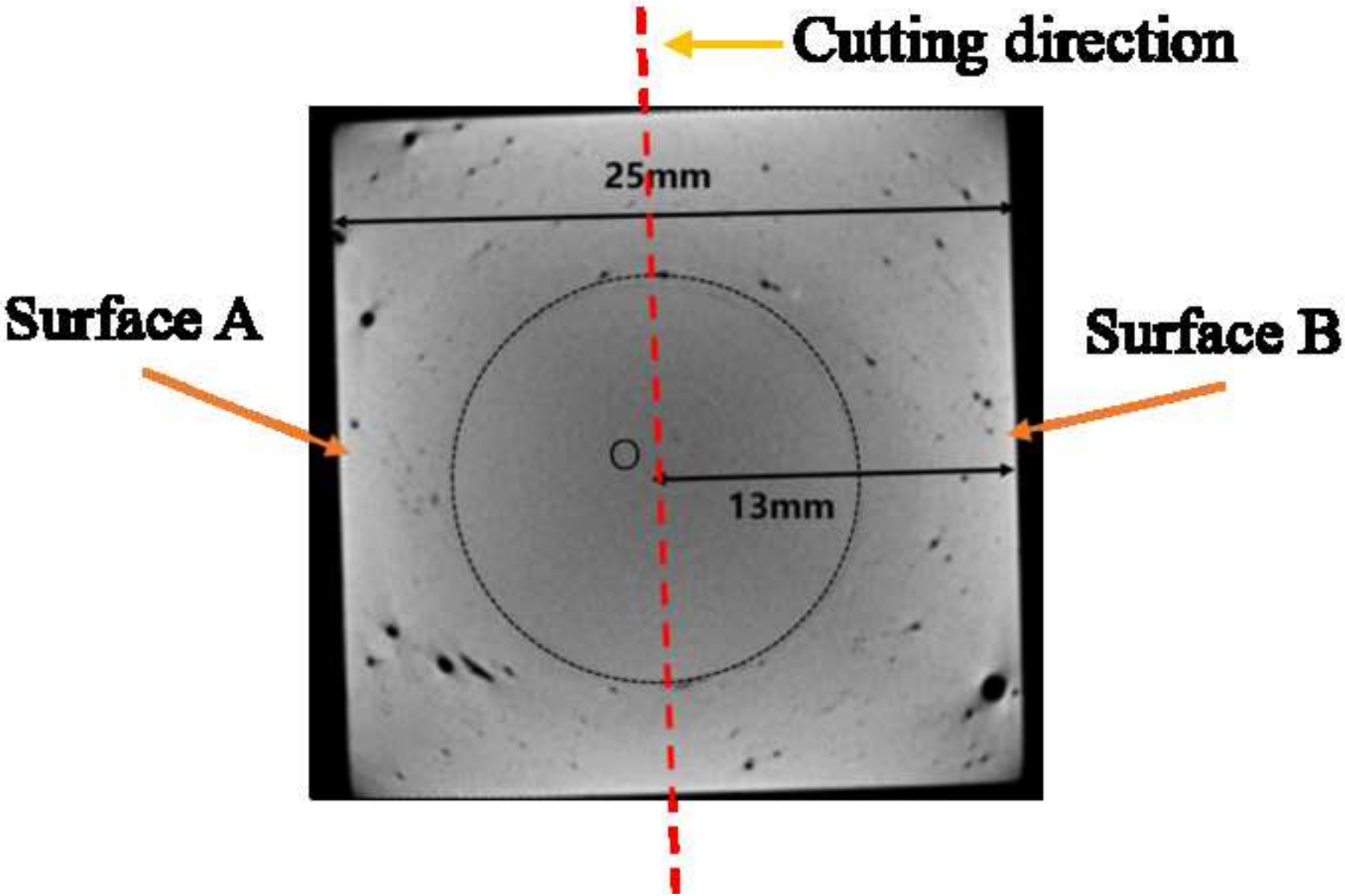

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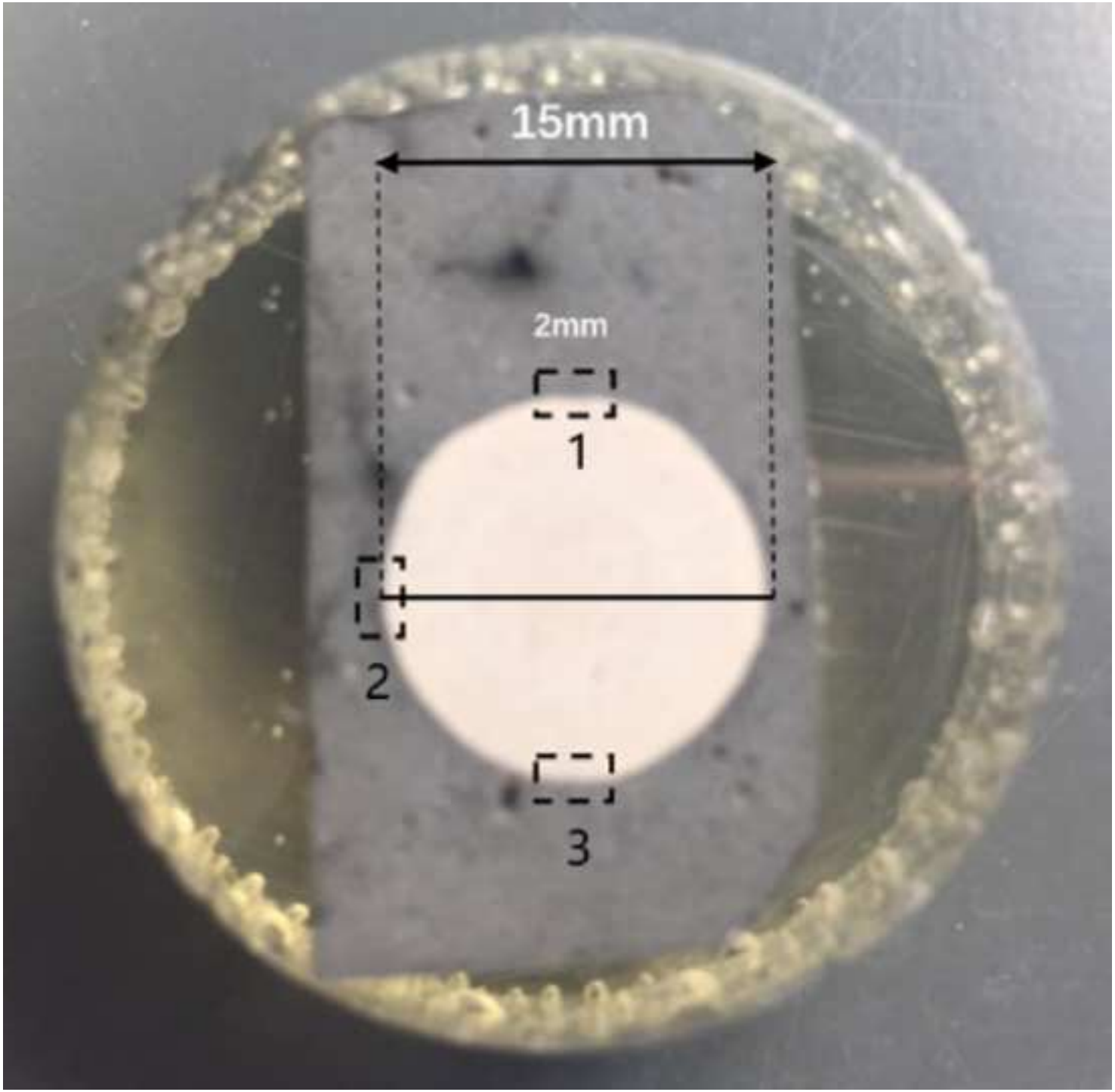

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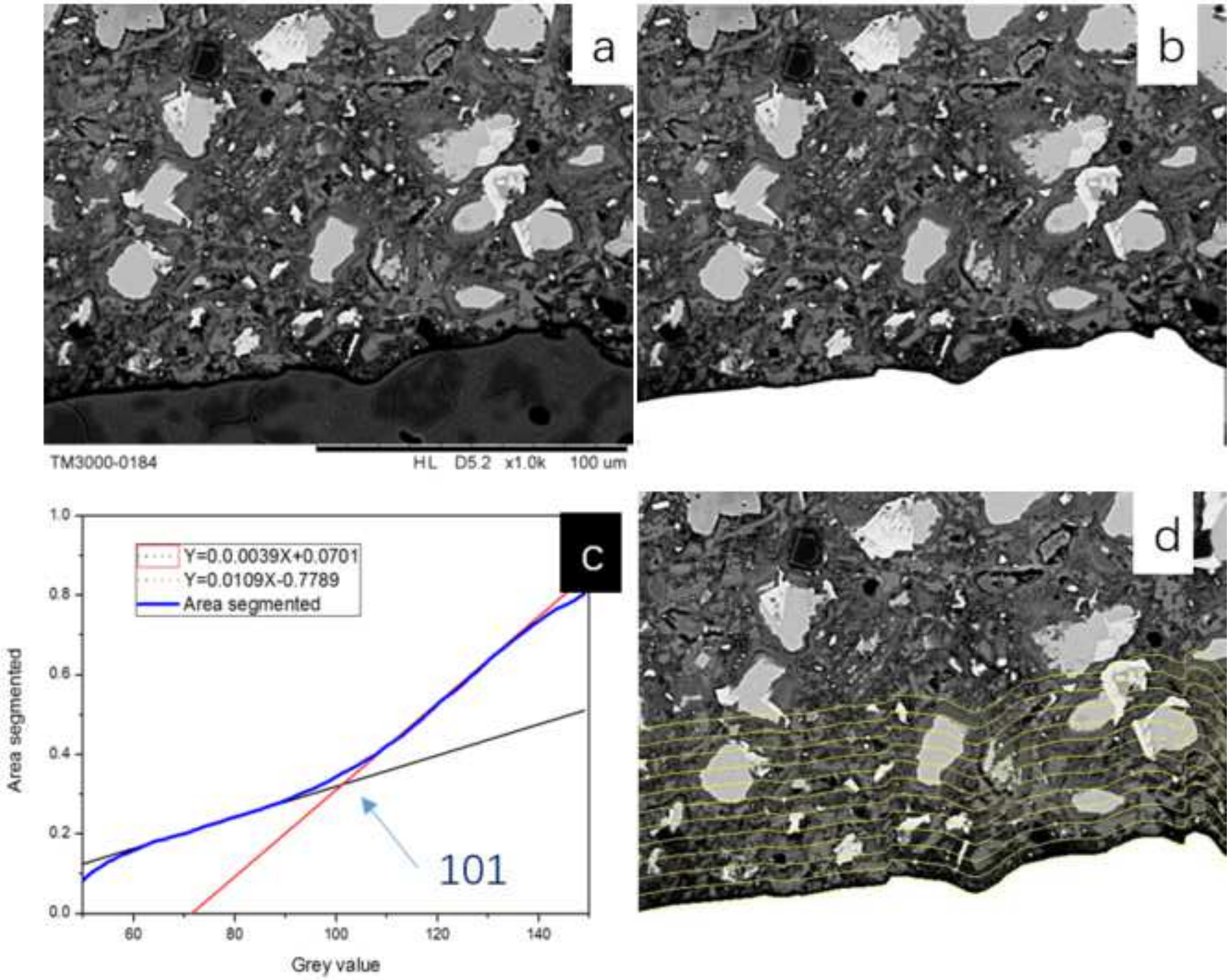

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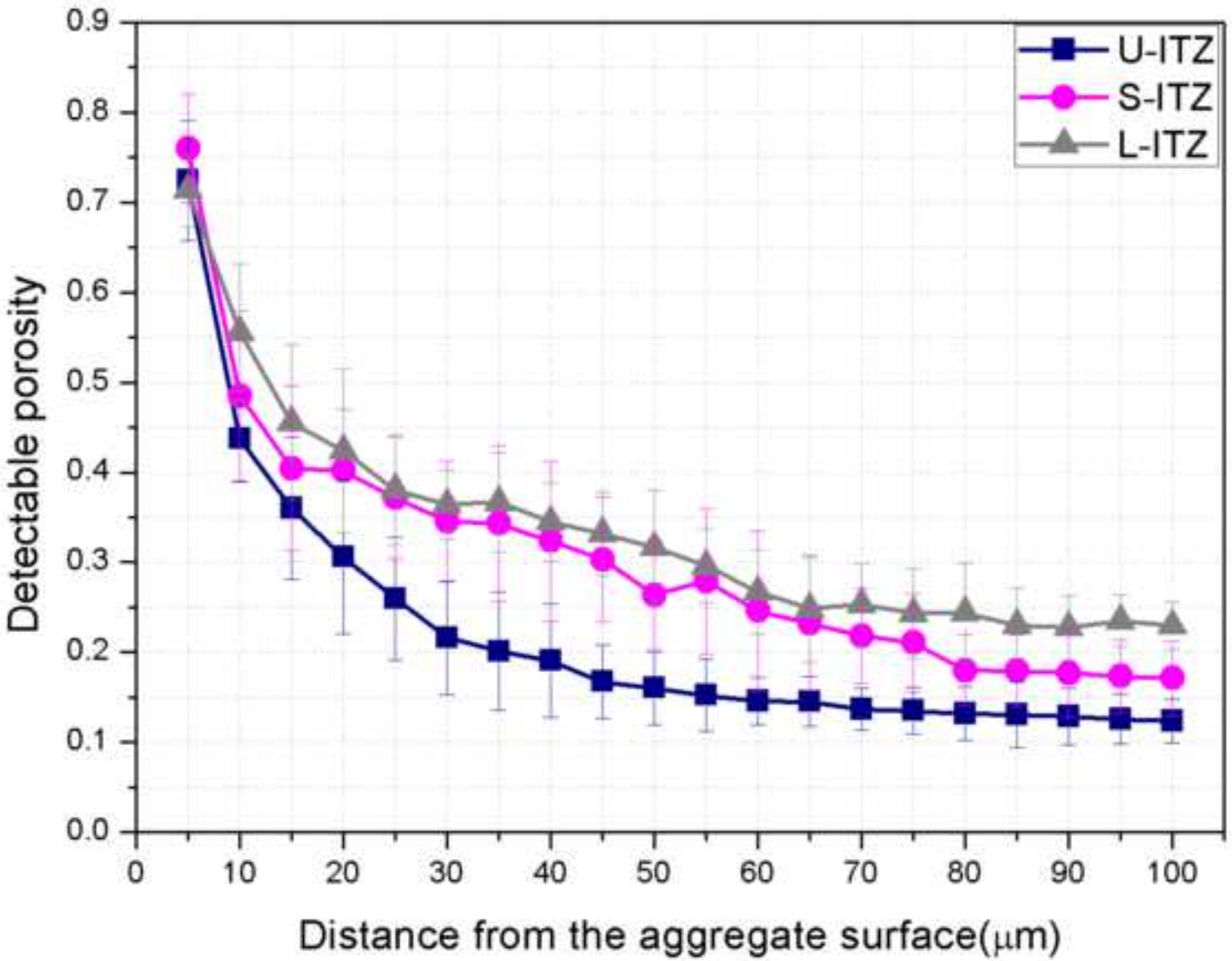

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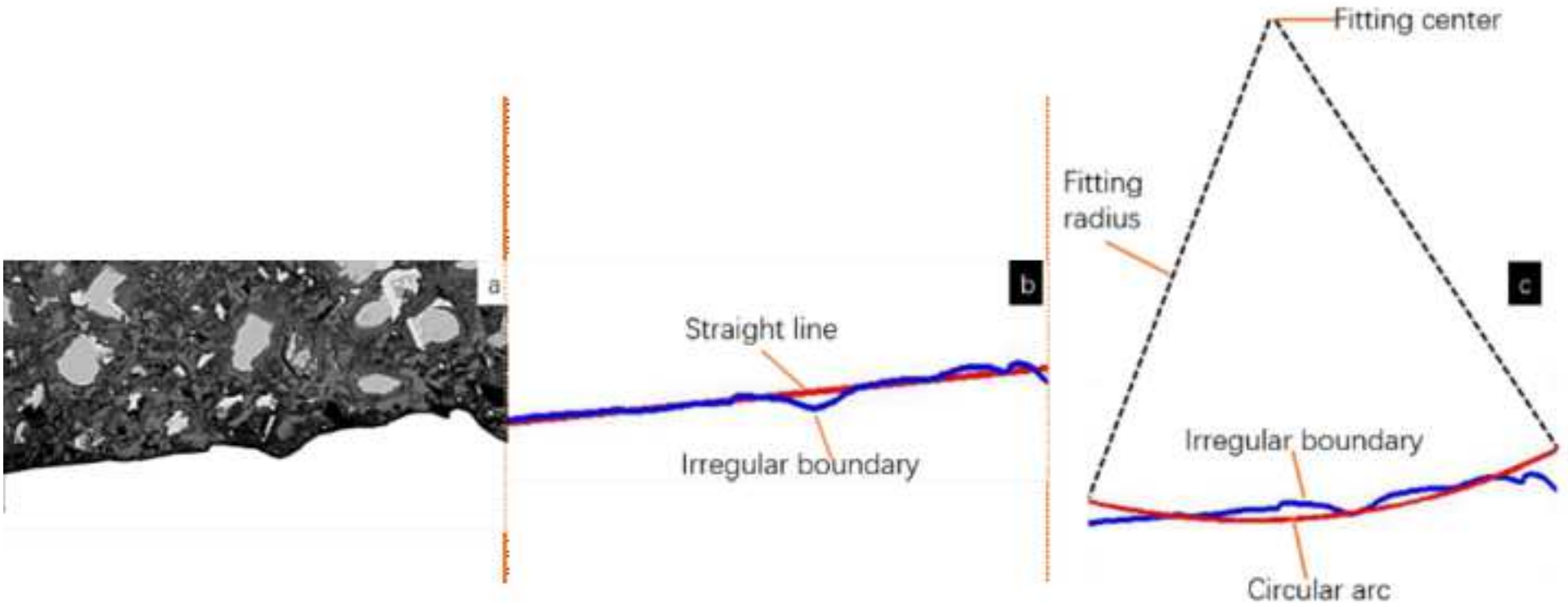

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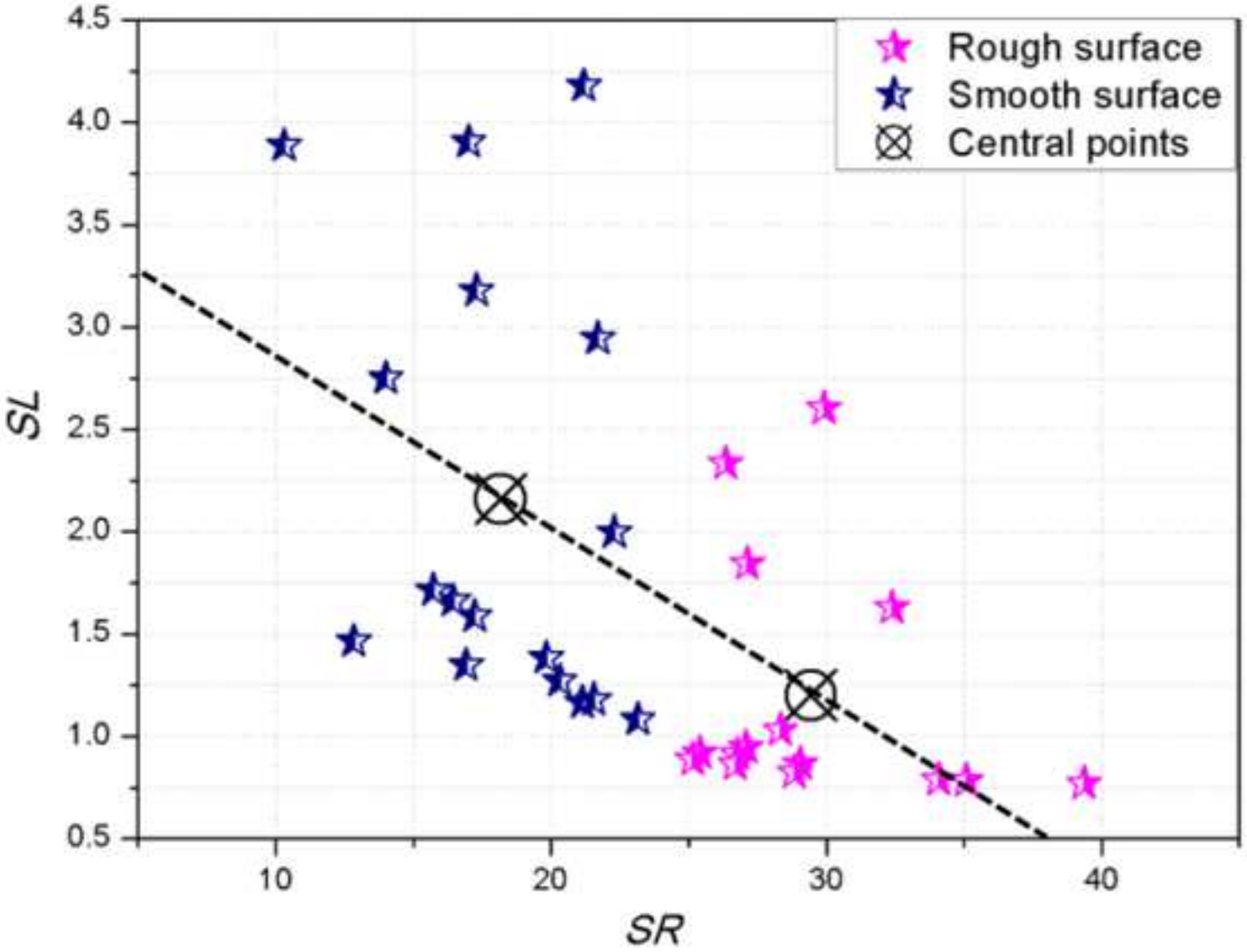

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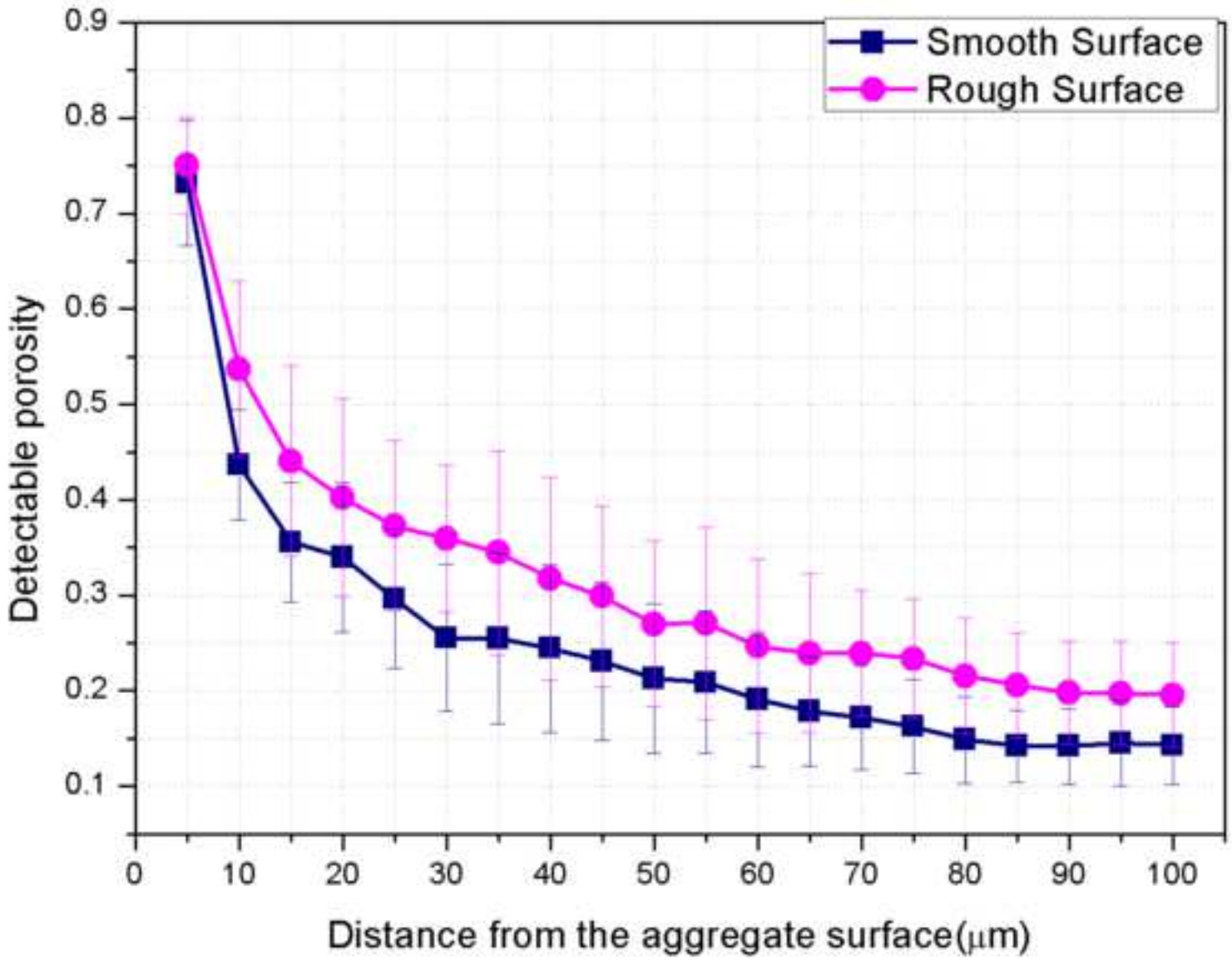

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Thank you for your letter and comments concerning our manuscript **JoVE60245** entitled "***Quantitative characterization of aggregate surface morphology on the characteristics of Interfacial transition zone (ITZ)***". The comments were helpful in revising and improving our paper and are listed below along with our responses. We have studied most of the comments carefully and have made changes that we hope meet with approval. In the paper, revised text is marked using Track Changes. Also, we made another highlighted manuscript no more than 2.75 pages.

We are looking forward to hearing from you soon!

Best, Kai

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