# Comparison of Quantitative Damage Characterization Methodologies

J.P.M. Hoefnagels [1], C.C. Tasan [2], L.C.N. Louws [1], M.G.D. Geers [1]

[1] Eindhoven University of Technology, Dep. Mechanical Engineering, Eindhoven, The Netherlands
[2] Materials Innovation Institute (M2i), Delft, The Netherlands

E-mail: j.p.m.hoefnagels@tue.nl

## 1. Introduction

Unpredicted failure in metals is of growing scientific interest, as it is observed for a number of metals that are becoming increasingly popular for sheet metal applications (e.g. advanced high strength steels, aluminum 6xxx series). These failures are believed to be triggered by ductile damage evolution. Numerical damage evolution models are being developed to precisely predict "safe" deformation limits in forming and service. However, these models require accurate input of damage evolution laws of these types of metals. Many damage characterization methods are available for this purpose, which were initially analyzed and classified in the pioneering work of Lemaitre [1]. Indentation-based damage quantification, which couples the degradation of either hardness or elastic modulus to the evolution of damage, was regarded as the most promising method [1] and, ever since, is used frequently in literature (see, e.g., Refs. [1]-[5]) and in industry. However, in previous work we showed that this approach has significant intrinsic flaws, for both hardness- and modulus-based damage quantification [6]. For increasing degree of deformation, both the harness and the elastic modulus not only decrease due to damage, but also either increase or decrease due to many other 'hidden' factors, such as strain hardening, grain shape change, texture development, indentation pile-up, etc. (see Fig. 1). This excludes indentation as a suitable method for accurate damage quantification [6]. Therefore, in this present work, a number of other common and/or promising damage characterization techniques are critically compared: the area fraction methodology, X-ray microtomography, local density measurement, and local elastic compression test.

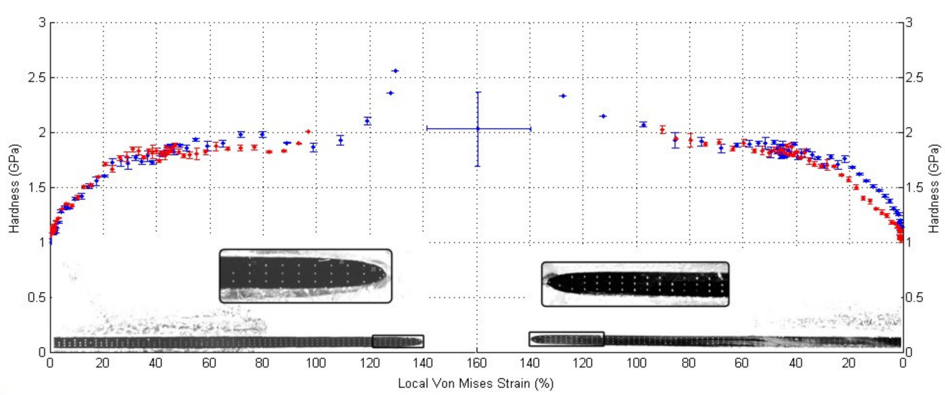


***Figure 1.*** *Indentation hardness as a function of local strain in IF steel [6], showing a monotonous increase of hardness with increasing strain. The expected decrease in hardness due to damage evolution is not observed, except maybe very close to the fracture surface (single data point with huge error bars at ε = 160%).*

## 2. Methodology

Non-homogeneous samples of aluminum 6016 (AA6016) are deformed up to fracture in a micro-tensile stage, while making real-time optical microscopy images of the deformation for digital image correlation analysis. Following fracture, 1mm$^3$ cubes are machined out by electro-discharge machining (Fig. 2(a)). The cross section of the remaining sample is mechanically polished using a carefully-tuned polishing protocol. These cross sections are analyzed with a Philips XL 30 ESEM-FEG and the resulting scanning electron microscopy (SEM) images are digitally processed (with free-ware software package 'ImageJ') for damage area fraction analysis (Fig. 2(b)). The 1mm$^3$ cubes, on the other hand, are used for the determination of the damage volume fraction, material density, and compression modulus. The damage volume fraction analysis is carried out using a high-resolution X-ray micro-tomograph (Nanotom®, spatial resolution of $\gtrsim$0.7μm). For high-sensitivity density analysis, the mass and volume of the machined cubes are precisely measured using, respectively, a sensitive mass balance and a surface profilometer (Sensofar PLμ2300© with 20x confocal microscopy objective). Finally, the cubed are compressed in the elastic regime (using a Kammrath-Weiss GmbH© deformation stage with 100mN load cell) to obtain the elastic modulus.

In addition, to gain more insight on the evolution of the compression modulus with void evolution, plane-strain elasto-plastic finite elements simulations of the elastic compression tests are performed (using finite elements simulation package MSC Marc Mentat©). The elasto-plastic material behavior is obtained from tensile experiments. Voids are modeled explicitly with a random spatial distribution (taking into account a minimum distance between the voids) and a random void size distribution that is sampled from the void size distribution function as obtained from experiments. A more detailed description of analogue explicit-void simulations that use the same representative volume elements (to simulate indentation tests, instead of compression tests here) is given in Ref. [6].

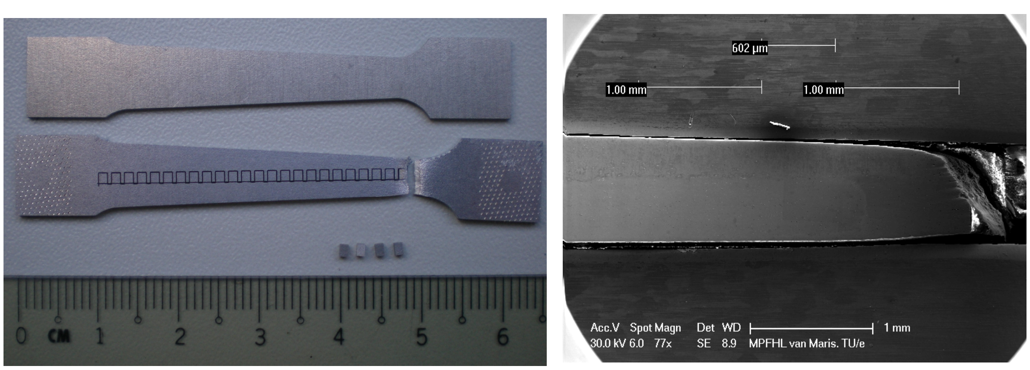


***Figure 2.*** ***(a)*** *The initial geometry of the AA6016 specimen used, shown together a specimen that was first strained up to fracture and subsequently electro-discharge machined to release the 1mm$^3$ cubes at different strain levels (also shown are four 1mm$^3$ cubes from another specimen), and* ***(b)*** *a SEM image of the mechanically polished cross section of the sample. The locations of two of the machined cubes are indicated by the 1.00 mm scale bars.*

## 3. Results

As explained above, first the specimens were deformed up to fracture and local strain maps were determined. These tensile tests induced significant damage formation with micron-sized voids in the aluminum alloy, as shown in Fig. 3. In the following, it is critically compared to which extent this damage can be quantified by the area fraction methodology, X-ray microtomography, local density measurements, and local elastic compression tests.

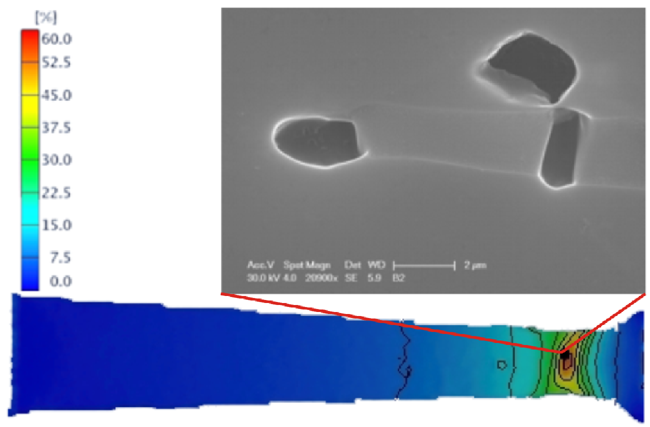


***Figure 3.*** *The local strain map as obtained from digital image correlation analysis. Significant damage evolution is observed (SEM image) towards the neck of the AA6016 specimen.*

### 3.1. Area Fraction Methodology

The most commonly-used methodology in the quantification of damage is through the determination of the area fraction of voids in SEM images. Although experimentation may be tedious, large amounts of results can be obtained easily. For the AA6016 specimen, the void fraction is observed to increase linearly with deformation (Fig. 4(a)), and a maximum void fraction of ~0.55% is observed for a strain of ~42%. An advantage of the area fraction methodology is that it also yields information on the (average) void size and distribution, and thus on the damage evolution. For instance, Fig. 4(b) shows that the number of voids saturates for large strain, indicating that a depletion of void initiation sites.

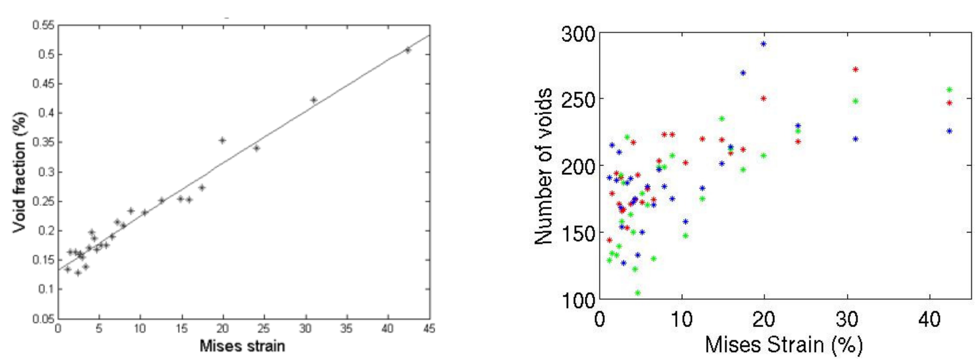


***Figure 4.*** ***(a)*** *Void fraction and* ***(b)*** *number of voids, calculated from SEM images via grey value 'thresholding'.*

Sensitivity analysis
It is easily seen that the results obtained with the area fraction methodology are very sensitive to experimentation, and in many cases it is not possible to measure absolute values at all. A separate sensitivity analysis, using more data than shown here, identified the main sources of systematic error being: the influence of specimen preparation method (even for careful specimen preparation, a minimum systematic error in the void fraction of ~0.2% is introduced), the manual setting of the grey value threshold (~0.1%), the damage gradients between different cross-sections (~0.05%), the influence of the SEM magnification used (~0.05%), and the assumption that the surface area void fraction equals the volume void fraction (only true for spherical voids). Apart from the obvious differences in absolute levels of void fraction, one should also be aware that these systematic errors can seriously distort the shape of the void-fraction–vs.–strain curve, as shown, e.g., in Fig. 5.

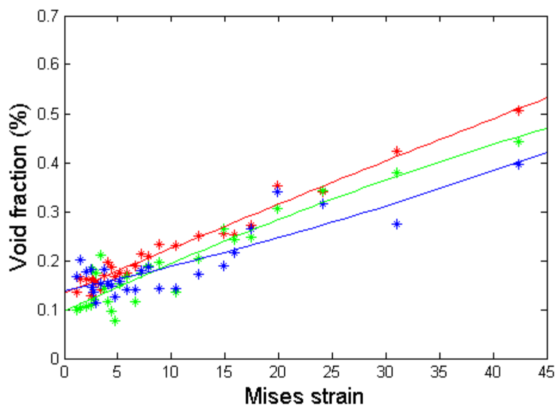


***Figure 5.*** *Void fraction measured at three different slices of the same sample, showing significantly differences not only in the absolute void fraction, but also in the shape of the curves.*

### 3.2 Volume Fraction Methodology

A typical result from X-ray microtomography of the volume fraction analysis is shown in Fig. 6. Here, the 3D reconstructed image of the sample from the neck is shown, together with a 2D cross-sectional slice close to the fracture surface, which clearly shows a number of voids of different size. The void fraction at this cross-section was analyzed to be ~1.5%. Besides the quantitative analysis, it is also possible to analyze the 3D morphology of the microvoids (not shown here.).

Sensitivity analysis
To analyze the sensitivity of the quantitative measurements from X-ray microtomography, a verification specimen was prepared, with a range of indents created by nanoindentation. The exact geometry of these indents is measured first using surface profilometry (Fig. 7(a)), and then compared with the X-ray microtomography methodology, to check the sensitivity of the latter. A number of X-ray settings have been analyzed to obtain maximum sensitivity for detecting the small indents (Fig. 7(b)). The optimized result is that indents up to a certain dimension are clearly visible (and are in the correct place). However, even for

optimized X-ray microtomography settings, the smallest void which is visible has a diameter of about 12 μm and a depth of 1.2 μm (Fig. 7(b)), which is much larger than the voxel size used (of ~0.7×0.7×0.7 $\mu m^3$). For smaller indents, the depth of the indent is approaching the voxel size, at which point voids become completely invisible. Another error is introduced by the manual action of setting the threshold to distinguish air (i.e. void) from material (in this case aluminum) in the gray-value histogram, which is very sensitive to a number of measurement parameters including the X-ray tube voltage, tube current, spot size, detector and specimen distance, etc. In fact, the inaccuracy in the threshold can easily introduce significant systematic errors in the void fraction of a factor of two (!), when the measurements are not carefully optimized and calibrated.

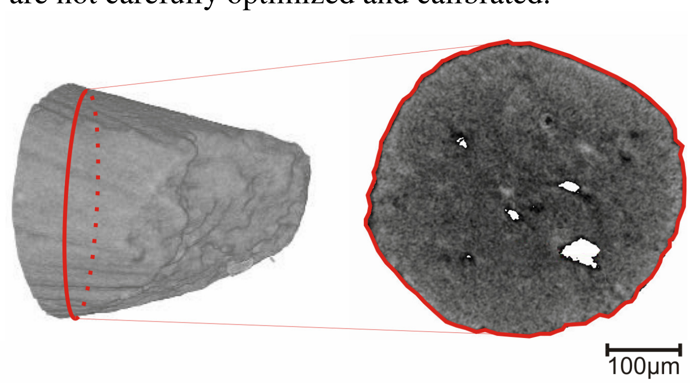


***Figure 6. (a)** 3D reconstruction of a specimen from the localized neck region obtained through X-ray microtomography, and **(b)** a 2D cross-sectional slice from the presented reconstruction, which shows some large voids close to the fracture surface.*

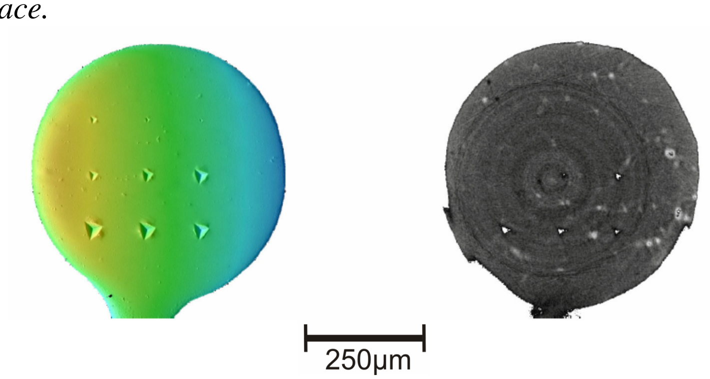


***Figure 7. (a)** Confocal microscopy profile of the nine indents of different sizes, and **(b)** the reconstructed slice from the X-ray microtomography measurement, using optimized operating setting, but still showing only the larger indents.*

### 3.3 Density Methodology

Density measurements are carried out by using a highly-sensitive mass balance to measure the mass, while the volume of the electro-discharge machined 1$mm^3$ cubes is measured very accurately – by sensitive surface profilometry – from the exact amount of liquid displacement due to cube submersion. Preliminary results are given in Fig. 8, which shows that the decrease of density due to the damage evolution is successfully captured in these measurements.

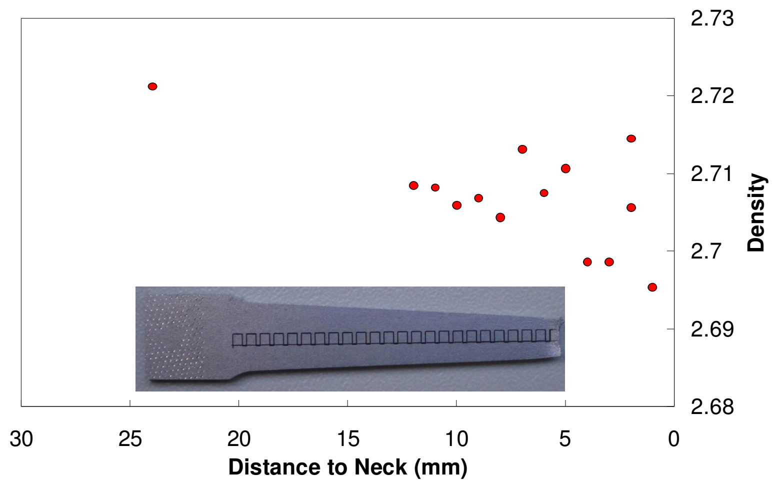


***Figure 8.*** *Results of density measurements of the electro-discharge machined cubes, as a function of distance to the fracture surface of the tensile specimen.*

<u>Sensitivity analysis</u>
In Fig. 8, clearly, considerable amount of scatter is seen. The measurement of mass can relatively easily be performed with an accuracy of 0.05%, which seems sufficient. The main challenge, however, is located in performing a highly-accurate measurement of the volume of the 1mm$^3$ cubes. The best candidate to obtain maximum volume accuracy is indeed the here-used methodology of liquid displacement, precisely measured using surface profilometry. However, volume accuracy remains a concern as systematic measurement errors are introduced through: thermal shifts in the environment, minute evaporation of the liquid medium (paraffin oil), and other factors such as errors introduces by stitching surface height maps. The methodology is currently being modified to decrease these error sources further.

### 3.4 Compression Methodology

Elastic compression tests to determine the modulus (Fig. 9) is a good probe for damage quantification, as the measurement of the modulus from compression is not limited by the fundamental pile-up problems seen for the indentation-based methodology (see Introduction or Ref. [6]). In addition, performing the compression test only in the elastic regime ensures that influences due to strain hardening and grain shape change are avoided, while the influence of texture development is at least significantly reduced and its remaining influence can be probed by elastically compressing the same cube over its three axes. Furthermore, finite element simulations show that compression modulus should be sensitive to even very small void fractions (Fig. 10 (a)).
It is noted that an elastic measurement of the local compression modulus is quite challenging due to the low forces and strains involved and the necessity to perform digital image correlation for strain measurement at the center of the cubes (Fig. 9). Nevertheless, in the preliminary tests on AA6016 cubes, reproducible loading/unloading curves are obtained, from which the modulus is obtained for different strain levels, as shown in Fig. 10(b).

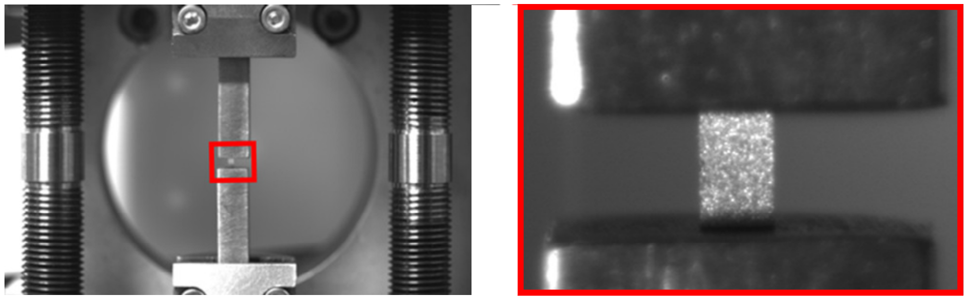

***Figure 9.*** *The compression tests are carried out in a micro-tensile stage with a 100mN load cell, by using two steel bars fixed in the clamps (left image) to compress the 1mm$^3$ AA6016 cubes (zoom-in image on the right).*

<ins>Sensitivity analysis</ins>

Whereas the reproducibility between loading/unloading curves of a single cube yields already a fairly large measurement inaccuracy in the modulus (error bars in Fig. 10(b)), the scatter seems to be significant enhanced when measured moduli of different cubes are compared (Fig. 10(b)). Still, a similar modulus–vs.–strain trend as found in the simulations may perhaps be visible. In fact, further analysis of the tested cubes revealed that each cube contained a so-called 'burr' on one of its corners, which is introduced by the electro-discharge machining process (Fig. 11). This burr is expected to have a significant influence on the measured modulus. Indeed, when the measured burr size of each cube is coupled with the modulus measurements, it is seen that cubes with relatively large burrs are the specimens (indicated by a black arrow in Fig. 10 (b)) that do not fit the expected modulus–vs.–strain trend. Currently, new specimen manufacturing methods with stricter geometrical tolerances are being considered. Still, also significant other measurement inaccuracies are active, e.g., due to sample misalignment, and load and strain sensitivity. As a more fundamental improvement to the methodology, we suggest to perform elastic compression tests, using a sensitive micro-indenter, on (an array of) sub-millimeter pillars (diameter of a few hundred micrometer) that are processed in, but still fixed to, the tensile specimen.

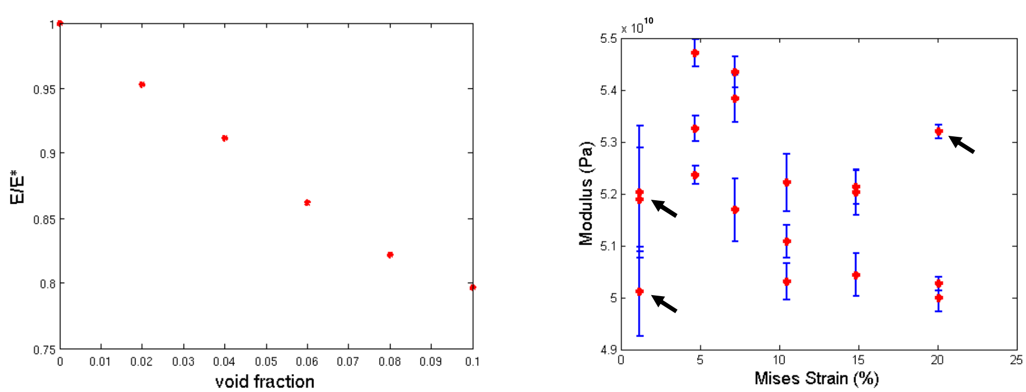


***Figure 10.*** ***(a)*** *Simulation results showing the decrease in relative compression modulus versus void fraction and* ***(b)*** *experimental results showing modulus with respect to local strains in the center of the cubes. The black arrows indicate those modulus measurements that are most influenced by the 'burr', see Fig. 11.*

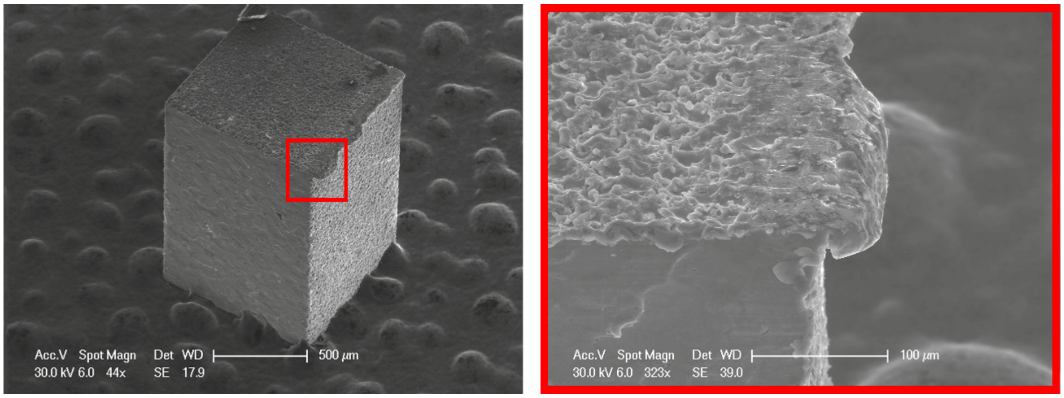


***Figure 11.*** *The 'burr' due to the electro-discharge machining causes scatter in the elastic compression tests.*

## 4. Conclusions

- The area fraction method is a common, seemingly practical method in quantifying damage. Our results show, however, that the method is very sensitive to many experimental parameters and hence should only be used for cases where high accuracy or quantification of absolute damage parameters is not necessary.
- The sensitivity of the volume fraction method is basically determined by the resolving power of the equipment used. However, even when excellent resolution and high signal/noise ratio is possible, the 'thresholding' process of voids and material may introduce a significant systematic error.
- The preliminary results shown here for the density methodology are promising. However, further improvement of the experimental noise in the local volume measurements is challenging.
- The elastic compression tests may perhaps show a decrease of modulus with increasing strain, as predicted in the simulations. However, a better specimen processing method is a requisite. Alternatively, an improved (mesosized-pillar-based) local compression methodology was suggested.

## 5. References

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