Study of Biaxial Stress Induced by Cosputtered Thin Molybdenum Silicide Films in Silicon Single-Crystal Substrates

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Abstract
Bending of 25 mm-diameter (100) silicon wafers produced by 100 nm-thick cosputtered molybdenum–silicon films has been investigated just after deposition, after rapid thermal annealing and after etching of the deposits. Values of biaxial stress in the films were determined by the use of a widely used relation. The Mo:Si ratio in the as-deposited films was 89:11. Radii of curvature of substrates were measured by a double-crystal X-ray diffractometer. Blank wafers with three widely different radii of curvature (33, 62 and 250 m) were chosen as specimens and their curvatures were taken into account in the determination of the value of biaxial stress in wafers with deposits. All the wafers were convex when viewed from the polished surface side on which the films were deposited. Values of \( \sigma \) for wafers with as-deposited films were in the range \( 3 \times 10^8 \) to \( 14 \times 10^8 \) N m\(^{-2}\) (tensile). Wafers with higher initial bending showed higher values of stress. Deposition led to degradation of perfection, as revealed by broadening of the diffraction curves and the contrast in the topographs. Rapid thermal annealing at 1273 and 1373 K (3-4 min) led to formation of the MoSi\(_2\) phase and to a notable relaxation of stress. The values of \( \sigma \) were in the range \( 1 \times 10^7 \) to \( 6 \times 10^7 \) N m\(^{-2}\) (tensile). The value of the stress was lowest for the blank wafer with smallest bending. Annealing also improved the degree of crystalline perfection of the silicon. Experiments performed after etching of the annealed specimens showed no significant change in the radii of curvature.

1. Introduction
Thin deposits on single-crystal substrates introduce biaxial stress (Bohg, 1978; Schwuttke & Howard, 1968; Glang, Holmwood & Rosenfeld, 1965) owing to the differences in the lattice parameters of the substrate and the film as well as to the differences in the thermal-expansion coefficients of the two materials. Phase transformations or chemical reactions may occur in films after deposition, leading to a change of volume and therefore to stress. The stress lowers the degree of crystalline perfection of the wafers and can result in a degraded performance or complete failure of the devices fabricated out of such crystals (Lal, Goswami, Wurfl & Hartnagel, 1990). Therefore, accurate determination of stress is of considerable importance.

The low resistivity of refractory metal silicides is a very desirable property for large packing density of integrated circuits based on silicon. A number of silicides such as those of molybdenum, palladium and platinum (Angillelo, D'heurle, Peterson & Segmüller, 1980) have been investigated as Ohmic contacts. Schwuttke & Howard (1968) were the first to investigate stress induced by molybdenum films in silicon substrates. However, they did not study molybdenum silicide. Murarka, Fraser, Retczyk & Sheng (1980) have measured stress owing to thin deposits of Mo\(_3\)Si, Mo\(_3\)Si\(_2\) and MoSi\(_2\). However, their silicon substrates had \(~300\ nm\)-thick oxide layers over which the silicides were deposited. We have investigated biaxial stress introduced by cosputtered Mo–Si films on (100) silicon single-crystal wafers. Annealing at elevated temperatures was used to synthesize MoSi\(_2\) films. We have also investigated wafers after plasma etching of MoSi\(_2\) films to determine whether the deformation produced by MoSi\(_2\) films in the wafers is elastic or plastic.

2. Experimental
2.1. Specimen
The specimens were (100) single-crystal silicon wafers (\( n \) type, \( \rho =5-7\ \Omega \ cm, \sim 25 \ mm \) in diameter and 300 \( \mu \)m thick). The wafers were polished on one side. They were prepared by the usual mechanical–chemical lapping/etching techniques employed to produce specimens for microelectronic device fabrication. About 100 nm-thick thin films of molybdenum–silicon were deposited by the cosputtering method (Wasan, 1978). The atomic ratio of Mo:Si was 89:11 as determined by Auger electron spectroscopy. For synthesis of MoSi\(_2\), the
wafers with deposition were subjected to rapid thermal annealing at 1073, 1273 and 1373 K for 3 and 4 min. The formation of the tetragonal MoSi₂ phase in the film was confirmed by powder X-ray diffraction. It was confirmed that no other phase was present even at the interface (within the sensitivity of the X-ray diffraction technique). The thickness of the silicide thin films was estimated by Auger depth profiling employing the Auger spectrometer (model PHI 90A). Argon ion sputtering was done at the rate of ~5 nm min⁻¹. The Auger profiling was carried out with an electron beam of ~100 nm with 3 kV as the accelerating voltage. The typical modulation voltage used was ~2 V. The calibration of the thickness was carried out with respect to the Al₂O₃ sputter profile. Talystep measurements (Taylor-Hobson, Leicester, England) were made on additional wafers over which a step was deposited simultaneously with the specimens. The results of these two measurements were in good agreement with each other. The thickness of the substrate wafers was measured by a digital micrometer having a least count of 1 µm (Mitutoyo, Tokyo, Japan).

2.2. Double-crystal X-ray diffractometer

A double-crystal X-ray diffractometer designed and developed in our laboratory was used in this study (Lal & Kumar, 1985). Fig. 1 shows a schematic diagram of the system. An X-ray beam from a fine-focus molybdenum source (sealed tube; Philips; 2 kW; 0.4 × 0.4 mm after foreshortening) was collimated with the help of a ~400 mm-long horizontal collimator having a 0.4 mm-wide vertical slit at one end. The collimated beam was diffracted from an asymmetrically cut (111) silicon single crystal in Bragg geometry. The asymmetry factor b, defined as (Kohra & Kikuta, 1968):

\[ b = \sin(\theta - \alpha)/\sin(\theta + \alpha) \]  

was approximately equal to 0.1. Here, \( \theta_B \) is the Bragg angle for the (111) diffracting lattice planes of a silicon single-crystal monochromator and \( \alpha \) is the angle between these planes and the surface of the crystal. The monochromator spreads the beam in the horizontal direction to a width of ~4 mm. The specimen was oriented for diffraction from (022) diffracting planes in symmetric Laue geometry and (+, -) setting of the diffractometer.

Biaxial stress is determined from the experimentally measured values of radii of curvature or bending of the substrates (Rozgonyi & Ciesielka, 1973). By using a high-resolution X-ray diffraction technique, we have measured the radii of curvature of specimen wafers. For this purpose, the change in the orientation of the diffraction vector \( \mathbf{g} \) for (022) in symmetrical Laue geometry was measured as a function of the linear position of the wafer as it was translated across the primary beam (Lal, Goswami, Wurfl & Hartnagel, 1990). For this experiment, as well as for diffractometry, the width of the exploring beam in the plane of diffraction was reduced to ~0.4 mm. If the wafer is bent spherically, the curve obtained by plotting the orientation of \( \mathbf{g} \) as a function of the linear position is a straight line. The plot will be a straight line even if the wafer was bent cylindrically with its generator perpendicular to the plane of diffraction. In general, a surface will have two principal radii of curvature. However, preliminary experiments with the (022) diffraction plane showed that the shapes of the wafers approximate to a spherical surface. The sign of the slope helps in determining the nature of bending (concave or convex) with respect to a given surface, generally the polished surface.

The biaxial stress is determined by using the well-known relation (Segmüller, 1982):

\[ \sigma = \frac{E}{6(1-\nu)} \left( \frac{t_s}{t_f} \right) \left( \frac{1}{R} \right). \]

Here, \( E \) and \( \nu \) are the elastic modulus and Poisson ratio of the substrate, respectively. In this investigation, we have used values of \( E \) and \( \nu \) for (011) and (100) of the silicon lattice. \( t_s \) is the thickness of the substrate, \( t_f \) is the thickness of the film and \( R \) is the radius of curvature of the wafer. The value of \( E/(1-\nu) \) for the (100) plane of the silicon substrate is 1.805 x 10⁶ N m⁻² (Brantley, 1973; Angillelo, D’heurle, Peterson & Segmüller, 1980). Values of elastic constants for other orientations of silicon are available in the literature (Brantley, 1973).

For taking into account the bending of the blank wafer, we have used the following relation to deduce the value of \( \sigma \):

\[ \sigma = \left[ \frac{E}{6(1-\nu)} \right] \left[ \frac{t_s^2}{t_f} \right] \left[ \frac{1}{R} \right] - \left( \frac{1}{R_0} \right), \]

where \( R_0 \) is the radius of curvature of the blank wafer.

Diffraction curves, topographs and curvature plots were recorded at the following four stages: (i) blank wafers, (ii) just after cosputtering of Mo–Si, (iii) after rapid thermal annealing and (iv) after plasma etching of the deposits. Stationary crystal topographs covered

![Fig. 1. A schematic line diagram of the double-crystal X-ray diffractometer developed at NPL and used in this investigation.](image-url)
a width of ~4 mm of the specimen in the horizontal plane, which was the plane of diffraction. To cover the entire volume of the specimen, a series of topographs was recorded on the same film. After each exposure, the specimen crystal and the film, which was rigidly coupled to it, were linearly moved across the X-ray beam by a distance of 4 mm (equal to the width of the exploring beam). The result of this experiment is a composite topograph comprising a series of six to seven topographs nearly touching each other.

3. Results and discussion

3.1. Determination of biaxial stress

Fig. 2 shows a typical curvature plot of a blank silicon wafer (sample 2). The plot is a straight line, showing that the specimen is bent along a spherical surface. As mentioned above, this observation can as well be understood in terms of a cylindrical bending. However, preliminary experiments with other diffracting planes exhibit similar bending, showing that the wafer is bent like a spherical shell. From the slope of this plot, the value of the radius of curvature has been determined as 62 m. This wafer was found to be convex in shape when seen from the polished surface on which Mo–Si film was afterwards deposited. The blank specimens used in this investigation were selected to have different radii of curvature \( R \). At one extreme, a highly bent wafer with a radius of curvature of only 33 m was selected, whereas at the other extreme was a wafer with low bending \( (R = 250 \text{ m}) \). However, all the wafers were convex in shape when viewed from the polished surface of the substrate.

Fig. 3 shows curvature plots of the wafer of Fig. 2, just after deposition (curve 2) and annealing (curve 3). For the sake of convenience of comparison, curve 1 is for the blank wafer (the same as Fig. 2). A comparison of the blank wafer plot (curve 1) with that of the same specimen after film deposition (curve 2) shows remarkable differences. Firstly, the shape of the wafer has changed. The convex polished surface has been transformed into a concave shape after deposition. Therefore, the stress is tensile in nature. The value of the radius of curvature is 58 m. The value of the stress obtained by using (3) comes out to be \( 9.04 \times 10^8 \text{ N m}^{-2} \) for this wafer. While calculating the value of \( \sigma \) we have measured the curvature of lattice planes, whereas \( R \) and \( R_0 \) in (3) relate to the physical surface of the specimen. The crystal surfaces and the nearest lattice planes made an angle of less than 0.5° with each other. The uncertainty due to this is insignificant and can be ignored.

We have studied the effect of initial bending of the wafers on the final stress values due to thin deposits. As mentioned above, one of the specimens was considerably bent \( (R = 33 \text{ m}) \). This specimen was found to be subjected to the highest level of stress, equal to \( \sigma = 13.73 \times 10^8 \text{ N m}^{-2} \). The least bent wafer \( (R = 250 \text{ m}) \) had the lowest value of stress, equal to \( 3.31 \times 10^8 \text{ N m}^{-2} \). The stress was tensile in all cases. We see that not only should one start with measurements on blank wafers, but...
wafers with large radii of curvature should be selected for film deposition to ensure low values of stress.

A general result of considerable significance has emerged from this investigation: that the value of the radius of curvature of the blank wafer and its sense (concave or convex) must be determined before the deposition of desired layers. As far as we know, this is being reported for the first time. If measurements are made only after films are prepared, as is generally done, one may deduce values of stress that are far from the true quantities. For example, in the case of Fig. 3, one can deduce the value of the stress immediately by putting \( R = 58 \text{ m} \) in (2). This comes out to be \( \sigma = 4.67 \times 10^8 \text{ N m}^{-2} \). However, this would be true only if the wafer before deposition was absolutely fiat, i.e., \( R_0 = \infty \). This value is almost half of the value obtained from (3). Since the wafer was bent from a convex to a concave shape, the value of the stress is expected to be high. We have experimented with a large number of wafers of silicon and other semiconductors like GaAs for determining stress due to oxides, nitrides and oxynitrides (Halder, Kumar, Lal, Kumar & Agnihotri, 1993) in silicon and metallicizations in GaAs (Lal, Goswami, Wurfl & Hartnagel, 1990; Goswami, Lal, Wurfl & Hartnagel, 1993).

No wafer is absolutely fiat. The bending ranges from an \( R \) of few tens of metres to an \( R \) of a few kilometres (in some cases only). Therefore, measurements of the curvature of blank wafers are essential to get data that are close to true values.

Wafers with cosputtered films were subjected to rapid thermal annealing at 1073, 1273 and 1373 K for varying lengths of time ranging from 30 s to 4 min. It was observed that the film is totally converted into the MoSi\(_2\) phase when subjected to 1273 K or more for 3 min or longer. Powder X-ray diffractograms revealed that no other phase was present after this treatment. Rapid thermal annealing produced remarkable changes in the stress level. Curve 3 of Fig. 3 shows a curvature plot of the wafer of Fig. 2 after annealing. The wafer has become convex (when viewed from the film side), although it had turned into a concave shell just after film deposition. Also, the bending has decreased substantially on annealing, as shown by the value of the radius of curvature. The value of the stress has been determined by the use of (3). It is \( \sigma = 2.63 \times 10^8 \text{ N m}^{-2} \) for this wafer. It is tensile in nature. The other wafers also showed similar results. The values of stress ranged from \( 1.0 \times 10^7 \text{ N m}^{-2} \) to \( 5.83 \times 10^8 \text{ N m}^{-2} \). For convenience of comparison, some typical values of the stress and radii of curvature are given in Table 1.

We have investigated our specimens after etching away the MoSi\(_2\) deposits produced by rapid thermal annealing to see whether the stress was elastic or plastic in nature. There was no significant change of radii of curvature of specimens 1 and 3 on etching. However, in the case of specimen 2, an increase in the radius of curvature is observed (from 156 to 382 m). From these results, it is not possible to draw a firm conclusion. However, qualitatively, these observations suggest that the substrate shapes are not significantly changed after etching. One has to keep in mind that, before etching, these wafers had been subjected to high-temperature annealing. Annealing at elevated temperatures is expected to produce a significant reduction in stress/strain, leading to a decrease in bending. We see that in all the cases there has been a significant increase in radii of curvature as compared to those in the blank stage. The effect of annealing also manifests itself in an enhancement of the degree of perfection of the substrate wafers, as will be seen in §3.2. One would have liked to see the effect of etching without interference from the changes produced by annealing; however, annealing is unavoidable as the MoSi\(_2\) phase is formed only when specimens are annealed at 1273 K for at least 3 min. Therefore, even though etching has produced no significant change in two wafers, the stress may still be elastic.

### Table 1. Some typical values of radii of curvature and biaxial stress in three specimens at different stages from the blank state to after rapid thermal annealing

<table>
<thead>
<tr>
<th>Sample</th>
<th>Radii of curvature (m)</th>
<th>Biaxial stress ( \times 10^8 \text{ N m}^{-2} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>33</td>
<td>Blank wafer</td>
</tr>
<tr>
<td>2</td>
<td>62</td>
<td>Blank wafer</td>
</tr>
<tr>
<td>4</td>
<td>250</td>
<td>Blank wafer</td>
</tr>
</tbody>
</table>

3.2. Effect of film deposition and subsequent annealing on crystalline perfection of substrates

We have also studied the effect of thin-film induced stress on the degree of perfection of the substrate wafers, the results of which are discussed below.

Fig. 4 shows a typical diffraction curve of a blank silicon wafer recorded with (022) diffracting planes. Two peaks due to \( K\alpha_1 \) and \( K\alpha_2 \) components of the characteristic \( K\alpha \) line are well resolved owing to dispersion caused by different lattice spacings of the diffracting planes of the monochromator and the specimen crystals. The observed angular separation matches the theoretically expected value derived on this basis (Pick, Bickmann, Pofahl, Zwoll & Wenzl, 1977). Each of the peaks is quite sharp with a half-width of \( \sim 11'' \). The half-widths of all wafers investigated by us were close to this value. The theoretical value of the half-width can be calculated on the basis of the plane-wave
dynamical theory (Batterman & Cole, 1964). For (022) diffracting planes and Mo $K\alpha_1$ radiation, the theoretical value of the half-width is $2.3''$. The observed half-width in Fig. 4 is larger than the theoretical value. A large part of the broadening is due to (i) the difference in the lattice parameters of the diffracting planes of the monochromator and the specimen and (ii) the large width of the exploring beam ($\sim4$ mm in the plane of diffraction) combined with the fact that the specimen is not absolutely flat, but bent as we have seen in the foregoing section. The broadening produced by a radius of curvature of $\sim100$ m of the specimen is $\sim4''$. We conclude that the specimen crystals are free of major defects like boundaries and a high density of dislocations. Therefore, these have a good degree of perfection.

Fig. 5 shows a typical composite topograph consisting of seven strips covering the entire volume of one of the specimen crystals. Observation of nearly uniform X-ray intensity in all the topographs reflects the high degree of perfection of the specimen (Tanner, 1976; Bonse, 1962; Bonse, Hart & Newkirk, 1967). However, two white bands (one broad and another narrow), approximately along the vertical direction, are seen in all the strips. This feature is an artefact also seen in the image of the exploring beam and therefore in all the topographs of different crystals. As we shall see later, the stress induced by deposits produces large changes in the topographs and the appearance of the white band does not affect the main results. No line defect is observed in this topograph. Also, the area around the mounting cylinders is uniform in intensity, showing the stress-free mounting of the specimen on the diffractometer. For details, reference is made to an earlier publication from our group (Lal, Goswami, Wurfl & Hartnagel, 1990). The results of topographic evaluation are consistent with the diffractometric results. Topographs of all other blank specimens were also similar to that shown in Fig. 5.

Deposition of Mo–Si films produced significant bending and degradation in the perfection of substrate crystals owing to biaxial stress at the substrate–film interface. The latter feature was reflected as a broadening of the diffraction curves and a change of contrast in topographs.

Fig. 6 shows a typical diffraction curve (curve 1) of a wafer having a thin Mo–Si film deposit. For convenience of comparison, the diffraction curve of the wafer in the blank state is shown as curve 2. It is seen that deposition of the thin layer has substantially broadened the diffraction curves. The half-width has increased from 11 to 20''. Also, there is a small decrease in separation between the peaks of $K\alpha_1$ and $K\alpha_2$ radiations. This is due to the stress induced by the deposit. It may be mentioned that the exploring beam intensity in the case of the two curves was not the same. It was much smaller for curves 2 and, therefore, the peak heights of curves 2 are much smaller. Similar results were obtained with all the specimens. The half-width values were in a narrow range: 20–25''.

Fig. 7 shows a typical composite topograph of a specimen with cosputtered Mo–Si film. The contrast in different strips shows clearly the presence of stress, particularly when this topograph is compared with the topograph recorded before deposition (Fig. 5). The stress distribution was studied in some detail with the help of the following experiment. Stationary topographs were recorded by orienting the specimen at five different angular positions on its Mo $K\alpha_1$ diffraction curve. Starting
from the $\theta < \theta_B$ side, these correspond to diffracted intensity values $0.5I_{\text{max}}$, $0.75I_{\text{max}}$, $I_{\text{max}}$, $0.75I_{\text{max}}$ and $0.5I_{\text{max}}$, where $I_{\text{max}}$ is the intensity at the peak position. These orientations are indicated by A, B, C, D and E on the $K\alpha_1$ curve of this specimen in Fig. 6. Fig. 8 shows a set of topographs recorded at the five points on the diffraction curve as mentioned above. A strong variation in contrast is observed from topograph to topograph. There is a region of high intensity whose location is dependent on the orientation of the wafer and shifts in its position from topograph to topograph are clearly seen. The contrast in these topographs is mainly orientational in character (Tanner, 1976).

A typical diffraction curve of a wafer after rapid thermal annealing is shown in Fig. 9. Its half-width is only 12" in comparison to ~20" just after deposition. It is observed that broadening of this diffraction curve is drastically reduced by 8" as a result of annealing. Therefore, we see that annealing not only converts the deposited film into silicide but also leads to significant improvement in the degree of crystalline perfection of the substrate.

Contrast in topographs recorded after annealing also supports the results of diffractometric evaluation. Fig. 10 shows a set of topographs recorded at different orientations around the diffraction peak as in Fig. 6. The intensity in Fig. 10 is fairly uniform in contrast to that in Fig. 8.

Fig. 11 shows typical diffraction curves of specimen 3 after etching. Curve 1 is for the blank wafer and curve 2 is for the same wafer after removal of the MoSi$_2$ film.
The value of the half-width of curve 2 is \( \sim 8'' \), which is lower than that of the half-width of the curve for the blank wafer \( \sim \sim 11'' \). This improvement is obviously a result of annealing of the wafers. Similar results were obtained in the case of other specimens.

The contrast in the topographs recorded after etching of all the specimens showed no significant change.

Murarka et al. (1980) have measured stress induced by Mo–Si films (with different Mo:Si ratios) in silicon substrates. The Mo–Si ratio of about eight of the films used in the present investigations had been covered in their work. However, their specimens had \( \sim 350 \text{nm} \)-thick oxide films sandwiched between the substrate and the Mo–Si films. Therefore, it is not possible to compare our results with those of Murarka et al. Nevertheless, we can consider the stress values that may correspond to Mo:Si ratios of our specimen. The value of stress reported by these authors is \( 5.1 \times 10^8 \text{ N m}^{-2} \). This is very high in comparison to the typical values given in Table 1. Indeed, in comparison to specimen 4, this value is more than 50 times larger. It may be pointed out that Murarka et al. had reported that, when the Mo:Si ratio is 4, clustering of molybdenum can be detected by X-ray diffraction. In the present work, we have not detected a separate molybdenum phase in our films even though the Mo:Si ratio was \( \sim 8 \). Further, it was reported by Murarka et al. that simultaneous formation of three intermetallic
phases MoSi$_2$, Mo$_3$Si$_2$ and Mo$_3$Si leads to lowering of stress by void formation. In the present study, it is shown that by careful selection of blank wafers the level of stress can be substantially reduced. Nevertheless, this comparison cannot be made in a strict sense, since the specimens are different.

Schwuttke & Howard (1968) reported values of the stress induced by molybdenum films in silicon wafers. By varying the temperature of the substrate during deposition, they could change the sense of the stress from compressive to tensile (Holmwood & Glang, 1965). The film thicknesses in their investigation were about 1 µm and substrates were 1 mm thick. However, since stress varies as $t_j^2/t_f$ [(3)], we cannot directly compare the values of stress in our specimen with those reported by Schwuttke & Howard. Also, the films in their study were pure molybdenum and not silicides as investigated in the present work. The values of stress reported were $2.0 \times 10^8$ N m$^{-2}$ (compressive) and $2.3 \times 10^8$ N m$^{-2}$ (tensile). The possibility of stress reduction to about $1.0 \times 10^7$ N m$^{-2}$ in the case of MoSi$_2$ films in our investigation is very satisfying.

4. Conclusions

The present paper reports the results of measurement of biaxial stress and crystalline perfection of silicon single-crystal wafers with Mo–Si cosputtered films before and after rapid thermal annealing. The main conclusions are as follows.

(1) The values of biaxial stress induced by as-deposited Mo–Si films lie in the range: 3.31–13.73 × 10$^8$ N m$^{-2}$. The stress is tensile in nature. A substantial degradation in crystalline perfection of the substrate is observed after film deposition.

(2) After rapid thermal annealing at 1273 K (or 1373 K) for 3 min or longer, the Mo–Si films are transformed into the MoSi$_2$ phase. The stress was reduced significantly. It was in the range 1.0 × 10$^7$ to 5.83 × 10$^8$ N m$^{-2}$. The crystalline perfection of the substrates also showed remarkable improvement.

(3) It has been shown that it is essential to take into account the bending of the blank wafer. Otherwise, the values and nature of the stress measured may be erroneous. This is one of the main highlights of the present investigation.

(4) Wafers with the lowest level of bending show minimum stress after deposition and annealing. The ratio between the lowest and the highest stress level can be as high as 58. Therefore, it is of the utmost importance to choose blank wafers with radii of curvature of ~250 m or more.

(5) The distribution of stress can be directly observed in high-resolution X-ray diffraction topographs. Similarly, diffraction curves give a good indication of deformation in the substrate crystal.

(6) From the study of wafers after etching of MoSi$_2$ films, it is not possible to draw a firm conclusion about the nature of the stress, owing to the unavoidable step of annealing of the specimens.

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