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A Method to Measure the Tensile Bond Strength Between Two Weakly-Cemented Sand Grains

Lawrence I. Knab and Nathaniel E. Waters

U.S. DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
(Formerly National Bureau of Standards)
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U.S. DEPARTMENT OF COMMERCE
C. William Verity, Secretary

NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
Ernest Ambler, Director

A Method to Measure the Tensile Bond Strength between Two Weakly-Cemented Sand Grains

by Lawrence I. Knab and Nathaniel E. Waters, Center for Building Technology, National Institute of Standards and Technology

ABSTRACT

A method to measure the tensile bond strength between two weakly-cemented sand grains was developed. Special microloading testing equipment was developed to measure the force required to pull apart two sand grains. To illustrate the method, bond strengths were measured in tension for six pairs of cemented sand grains. A wide range in the bond failure stress occurred and was attributed primarily to (a) difficulties in identifying and measuring the actual bond failure surface area and, (b) eccentricity in the specimens during loading. The method developed is seen as a starting point and can be used as a basis for further development. Improved techniques need to be developed to identify and measure the actual bond failure surface area and to reduce, or at least measure and account for, the eccentricity introduced.

Keywords: bond; sand grains; strength; tension; test method; weakly cemented

1. INTRODUCTION

A research effort investigating the deformation characteristics of weakly-cemented sand grains at the sand-grain level was being sponsored by the Air Force Scientific Office of Research (AFOSR) and was being conducted by the Air Force Weapons Laboratory (AFWL). An important factor in the investigation is the relationshi of bond behavior (soil grain/matrix interface) to the macroscopic response of the overall sample. The determination of the bond failure mode and bond strength between two weakly-cemented sand grains tested in tension, compression, and torsion was needed. The National Institute of Standards and Technology (NIST) was requested by AFWL to investigate the tensile bond strength and behavior. Since there was no known test method for measuring the tensile bond strength of such specimens, a test method had to be developed.

2. PURPOSE AND APPROACH

The purpose of the NIST research was to develop a method to measure the tensile bond strength between weakly-cemented sand grains. ("Weakly-cemented" refers to the use of a cement paste with a high water/cement ratio). Special microloading testing equipment was developed to measure the force required to pull apart two sand grains. To illustrate the method, preliminary bond strengths were measured in tension for six pairs of cemented sand grains.

3. TEST METHOD AND SPECIMENS

3.1 Test Frame

A test frame was made as shown in figure 1. It consisted of upper and lower square stainless steel rods, 1/2 in. on a side, held together at their ends by threaded rods and spacers. The rods were spaced 7/8 in. apart. Six sets of holes were drilled in the stainless steel rods so that the holes were aligned and were slightly greater in size than the 0.029 in. diameter stainless steel wire to which the sand grains were attached and the load applied. The holes provided concentric guides for the wires but did not prevent the wires from moving freely. One sand grain was adhered to the end of the upper wire using an epoxy adhesive and the other similarly adhered to the end of the lower wire. The two sand grains were then bonded together with cement paste and, after curing over water, were pulled apart in tension.

3.2 Preparation of Wire and Adhering of Sand Grains to Wire

Stainless steel wire and stainless steel square rod was used to prevent rusting (during curing over water) which could cause increased friction between the wire and the walls of the guide holes. The wire was straightened prior to using. The lengths of the top and bottom pieces of stainless steel wire were 1 1/8 in. and 2 1/2 in., respectively. Before adhering the sand grains, the end of the wire to which the sand grain was to be adhered was sanded until flat and then immersed in hydrochloric acid(labelled "HCL Approx. 37 %") for two minutes and then dried. The acid treatment was used to improve the bond of the epoxy to the wire by cleaning and etching the end of the wire. The wires to which the sand grains were to be adhered were held vertically in a holder which could withstand oven-curing temperatures for the epoxy[a,b]. Using a sewing needle and a microscope, the epoxy was applied only to the wire and, using tweezers, the sand grain was carefully placed on top of the epoxy. The epoxy, which held the sand grains to their wires, was then heat cured at about 70°C for two hours (with the wires still in their holder). The oven used was not a circulating air type because the air flow could have blown the sand grains from the wires before the epoxy cured. Use of the epoxy and the procedures developed were satisfactory and resulted in no bond failures between (a) the epoxy and the wire and (b) between the epoxy and the sand grain, in the tests

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[a] The epoxy was 3M-1838 B/A manufactured by the Adhesives, Coatings, and Sealers Division of the 3M Company, St. Paul, MN.

[b] Certain manufacturers' names, and names of commercial equipment, instruments, and materials are identified in this report to adequately specify the experimental procedure. Such an identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the equipment, instruments, or materials identified are necessarily the best available for the purpose.

reported in table 1. Use of other procedures and materials (e.g., quick-setting epoxy) were unsatisfactory in some cases, where the bond strength between the epoxy and the sand grain, or the epoxy and the wire, was less than the bond strength of the sand grains held together by cement paste.

After placing the wire with the adhered (with oven-cured epoxy) sand grain in the (disassembled) stainless steel frame, an approximate 90° bend was made in the end-most 1/4 in. length of the upper wire and in the end-most 3/8 in. length of the lower wire to: (i) cause the end of the upper wire to bear against the stainless steel rod and not move when loaded, and (ii) serve as a hook for the loading device for the lower wire (see below). The stainless steel frame was then assembled and the sand grains bonded together with cement paste.

3.3 Bonding and Curing of Sand Grains

A cement paste was hand mixed using a water/cement ratio of 2.0 (50 g water to 25 g of a "Type I-II portland cement" provided by AFWL). The paste was initially mixed for 3 to 4 minutes but, because of cement settling to the bottom of the mixing bowl, would have to be remixed periodically throughout the application of the paste to the sand grains ("No. 10 Silica sand" used as received from AFWL). The stainless steel frame containing the sand grains was placed over water in a curing pan while the paste was applied so that they would not have to be moved during curing. The paste was applied using a modified spatula, with a small spoon-shaped tip with an elliptical-shaped top having long and short dimensions of about 0.12 in. and 0.06 in., respectively, and an average depth of about 0.04 in. Using a microscope for viewing, the paste was applied to both grains and then the grains were butted together, except for two cases (Specimens 2 and 6, table 1) where the nearest edges of the two grains were separated about 1/3 of a sand-grain diameter, respectively (sand-grain diameters used in all six specimens varied from about 0.050 to 0.065 in.). After applying the cement paste to six pairs of sand grains, the curing pan was covered without disturbing the stainless-steel frame and the freshly-bonded sand grains. Prior to being tested, the cemented sand grains were cured 14 days in a moist environment over water[c] at room temperature.

3.4 Tensile-Bond Testing of Sand Grains

As shown in figure 1, glass centrifuge tubes were used as loading devices. Wire was used around the top of the tubes to attach them to the bent end of the lower wire. The sand-grain specimens

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[c] Preliminary tests of sand grains bonded together with cement paste (water/cement = 2.0) showed that (a) curing in air for 3 days resulted in essentially zero bond strength (failure load of about 0.2 g, or less), and (b) curing over water for several days resulted, in some cases, in relatively large failure loads (e.g., one specimen failed at 227 g at 4 days and another failed at 211 g at 5 days).

were loaded by first hanging the lighter centrifuge tube (26.4 g[d]) on the bent wire, and then slowly adding water with a syringe. The 3.66 in.^{3} (60 ml) syringe had a "Luer-lok" tip fitted with a reusable 13-gauge, 4 in. long stainless steel, "Luer-lok" needle. If the tube became filled with water before failure, the tube was removed, the water emptied, and sufficient 1/4 in.-diameter steel balls were then put into the tube to approximately equal the weight of the water. The tube was then rehung on the specimen and water was added (with a syringe) to the tube and the steel balls. If the tube again became full of water, the above procedure was repeated, with more steel balls added to approximately equal the weight of the water emptied, until failure occurred. When the load in the lighter centrifuge tube reached the approximate weight of the empty heavier centrifuge tube (160.6 g), the heavier centrifuge tube was substituted and the procedure of adding water and steel balls was repeated as given above. To prevent the loss of any water during loading, a beaker was stationed under the centrifuge tube to catch it when the specimen failed. Failure load was measured by weighing the centrifuge tube and its contents. All six specimens were tested at approximately 40 percent relative humidity and within about 2 hours after removing from their moist curing in water vapor. The order of testing the specimens is given in table 1.

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[d] Units of grams are used rather than pounds because the load was applied and measured in grams.

There was, undoubtedly, some eccentricity introduced during testing of the sands grains. Possible contributing factors include out-of-straightness of wires, small horizontal movements of the upper and lower wires relative to each other, sand grains not centered on the ends of their wires, out-of-roundness of the sand grains, out-of-alignment of sand grains when they were bonded together with cement paste, and variations in the volume and thickness of the paste applied to the sand grains.

4. TEST RESULTS AND DISCUSSION

Table 1 presents the results for the six specimens tested, including the failure load and very rough estimates of the bond failure stresses, based on estimated bond failure surface areas. Each specimen tested consisted of a pair of sand grains. Table 1 lists the failure stress based on the estimated fracture surface as observed on the sand grain adhered to the upper wire, and also on the sand grain adhered to the lower wire.

4.1 Failure Mode

The failure surfaces of all specimens included, to varying degrees, the paste/sand grain interface. The remaining failure surface (in addition to the paste/sand grain interface) occurred in the paste; there was no observed failure of the sand grains (see figures 2 to 21). In addition, there was no failure of the interface between: (i) the epoxy and the wire,(ii) the epoxy and the sand grain, and (iii) the paste and the epoxy (as far as could be determined, the failure surface of the paste was confined to the sand grain surface and not to the epoxy bonded to the sand grain surface). Figures 2, 5 to 7, 9, 12 to 15, 18, 19, and 21 show the estimated bond surface failures, based on a planar area and with a 90°-viewing angle (viewed at approximately 90° to the failure surface), for the six specimens tested (table 1). The 90°-viewing angle was used to estimate the failure surface areas in table 1. In addition, a 0°-viewing angle (side view) and 45° viewing angle (viewed at approximately 45° to the failure surface) are shown in figures 3, 4, 8, 10, 11, 16, 17 and 20 for selected specimens.

The failure surface area of specimen 2 as viewed on the upper wire (figure 7) appeared to contain some relatively large porosity.

4.2 Uncertainty in Estimation of Bond Failure Surface Area

The estimated bond failure surface area was determined as follows.

(i) The perimeter of the failure surface was estimated by viewing at a 90° angle to the actual failure surface, using a light microscope. This perimeter was then marked on a photogragh of the failure surface (see figures showing the 90° viewing angle).

(ii) The length of a straight line from one side of the perimeter to the other was measured by viewing the actual failure surface with a 20-power machinist's microscope. The line length was read in 0.0001 in. units using a micrometer, mounted to the base of the microscope. Then the length of a similar line[e], at 90° to the first, was measured. The lengths of the two lines were then measured on the photograph. The relationships of the two line lengths on the actual failure surface to the corresponding line lengths on the photograph were used to obtain an average magnificati (see magnification scales on figures showing 90° viewing angle).

(iii) The planar surface area inside the perimeter of the failure surface, as it appeared on the photograph, was determined by overlaying the photograph with transparent graph paper (10 squares to the inch). Using the planar surface area from the photograph and the magnification[f], the planar area inside the perimeter of the actual failure surface as observed under the microscope was determined.

The uncertainty in determining the planar surface area inside the perimeter by using graph paper was considered insignificant compared to two major sources of uncertainty in estimating the

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[e] The two lines typically intersected near the centroid of the failure surface.

[f] That is, the magnification was used to determine the relationship between a unit area on the photograph and the corresponding planar area on the actual failure surface.

failure bond surface area:

(1) the assumption that the failure surface was planar, and

(2) the uncertainty in determining the perimeter of the fracture surface. That is, it was difficult to discern whether exposed sand-grain surface areas adjacent to evident failure surfaces were (a) part of the paste which formed the bond between the sand grains and had resisted the tensile pulling of the sand grains or (b) the result of paste which originally had coated the sand grain surface and spalled off during testing but had not participated in resisting the tensile pulling force. (When applying the cement paste to the sand grains, it was not possible to prevent some of the paste from adhering to portions of the sand grains where the adhered paste was unlikely to participate in resisting the applied pulling force.)

Because of these two major sources of uncertainty, and the additional uncertainty of the effects of eccentricity, the calculated failure stresses are considered very rough estimates of the actual failure stresses.

4.3 Estimation of Failure Stress

There was a wide range in the estimated failure stress values in table 1, with values ranging from a failure resulting from placing the 26.4 g loading tube on the specimen (Specimen 4, less than or equal to 44 psi) to 579 psi (Specimen 2). The very large variability in the stress values was attributed primarily to the effects of eccentricity and the substantial uncertainty in determining the bond failure surface area (see above). Ideally, the two estimated failure stress values for each specimen (one estimate based on the sand grain adhered to the upper wire and the other based on the sand grain adhered to the lower wire) should be the same. However, the ratio of the larger to smaller stress (equal to the ratio of the larger to smaller planar surface area) for each specimen ranged from 1.3 to 1.8. This large uncertainty was attributed to the substantial uncertainty in determining the bond failure surface area.

With Specimens 1, 3, 4, 6, and possibly 5 (see (ii) below), the larger of the two estimated stresses for each specimen was considered to be an upper bound (highest possible stress that could be obtained for the particular specimen, excluding eccentricit effects) because:

(i) the assumption of planar failure surfaces (curved surface would result in more area and less stress) and,

(ii) the higher stresses are based on well-defined failure surface perimeters which were considered to result in the minimum possible failure surface areas. That is, if errors were made in identifying the failure surface, they were most likely due to omission of a portion of the failure surface, giving too high a stress. With Specimens 1, 3, 4, and 6 (table 1), the smaller failure surface areas (mostly paste and approximately concave in shape - figures 2, 9, 13, 19) were better defined than the corresponding larger failure surface areas (approximately convex in shape - figures 5, 12, 14, and 21). With Specimen 5, the failure surface area (figure 15) associated with the higher stress was well defined but only slightly better defined than the corresponding larger failure surface area (figure 18).

The presence of eccentricity during testing would most likely decrease the observed failure loads shown in table 1 and thus, if the effects of eccentricity were eliminated, the failure loads and the associated failure stresses would most likely increase as compared to those given in table 1.

5. SUGGESTIONS FOR IMPROVING THE METHOD

The method as developed had a number of experimental difficulties. The following suggestions are offered to help overcome these difficulties in future research.

1. Identification of the fraction of the cement paste which resists the tensile pulling force is needed. High speed photography probably could be used to capture the failure sequence and to better

define the actual failure bond surface.

2. Documentation and quantification is needed of the eccentricity introduced during pulling apart of the sand grains. Photographic documentation of the sand grains for the following stages should help quantify the eccentricity:

(i) prior to their being bonded with cement paste, to show the eccentricity caused by the out-of-straightness of the wires, and by the sand grains not having been centered on the ends of the wires
(ii) prior to pulling apart (after curing), to show the eccentricity caused by out-of alignment of the sand grains when they were bonded together with cement paste, and
(iii) just after testing, with the two sand grains touching each other, to show the relative orientation of the failure bond surfaces on the sand grains.

Also, the 7/8 in. spacing between the square stainless steel rods (Section 3.1) should be decreased, if possible, to reduce the effects of eccentricity caused by out-of-straightness of the wires. It is believed that this dimension could be reduced while still permitting the application of the cement paste, which bonds the two grains together.

Also, if it can be found, the use of flexible (e.g., braided) stainless steel wire, instead of the stiff stainless steel wire used, should reduce the eccentricity introduced.

3. The curvature of the bond failure surfaces should be taken into account when determining their surface area. Potential methods include scanning electron microscopy using secondary electron imaging to depict the topography of the failure bond surface and image processing to determine the surface area and porosity of both planar and curved surfaces.

6. CONCLUSION

The method developed for determining the tensile bond strength between two weakly-cemented sand grains is seen as a starting point and can be used as a basis for further development. Improved techniques need to be developed to identify and measure the actual bond failure surface area and to reduce, or at least measure and account for, the eccentricity introduced.

Table 1. Failure Loads and Estimated Bond Failure Surface Areas and Stresses for Tensile Bond Tests[a]

Specimen[b] Number	Failure Load		Estimated[c] Surface Area and Failure Stress Based on Sand Grain Attached To: Lower Wire		Upper Wire	
	(g)	(lbf)	Area(in^2)	Stress(psi)	Area(in^2)	Stress(psi)
1	133	0.293	0.001277	230	0.00203	145
2	306	0.675	0.001163	579	0.00168[d]	402[d]
3	53.5	0.118	0.000642	184	0.00114	104
4	≤ 26.	0.057	0.000827	≤ 69	0.00129	≤ 44
5	133	0.293	0.000527	556	0.000676	433
6	140	0.309	0.000847	364	0.00125	246

[a] Failure surface area and stress are very rough estimates.

[b] Specimen number designates order of testing; "1" = tested first, etc.

[c] Cement paste was applied to both sand grains and then the grains were butted together, except for Specimens 2 and 6, where the nearest edges of the grains were about 1/3 of a sand-grain diameter apart. Sand grain diameters varied from about 0.050 to 0.065 in.

[d] Failure surface had a vertical portion, with an area of about 0.000891 $in.^2$ which, if added to the horizontal planar area of 0.00168 $in.^2$, results in an estimated failure stress of 263 psi. (At least part of the vertical portion chipped off prior to when the picture (figure 7) was taken)

Figure 1. Test Frame Showing a Failed Specimen with Glass Centrifuge Loading Tube Containing Steel Balls and Water.

Figure 2. Specimen 1, Sand Grain Adhered to Lower Wire, Estimated Planar Bond Failure Surface Area (Used in Table 1) Shown By Enclosed Area, 90°-Viewing Angle.

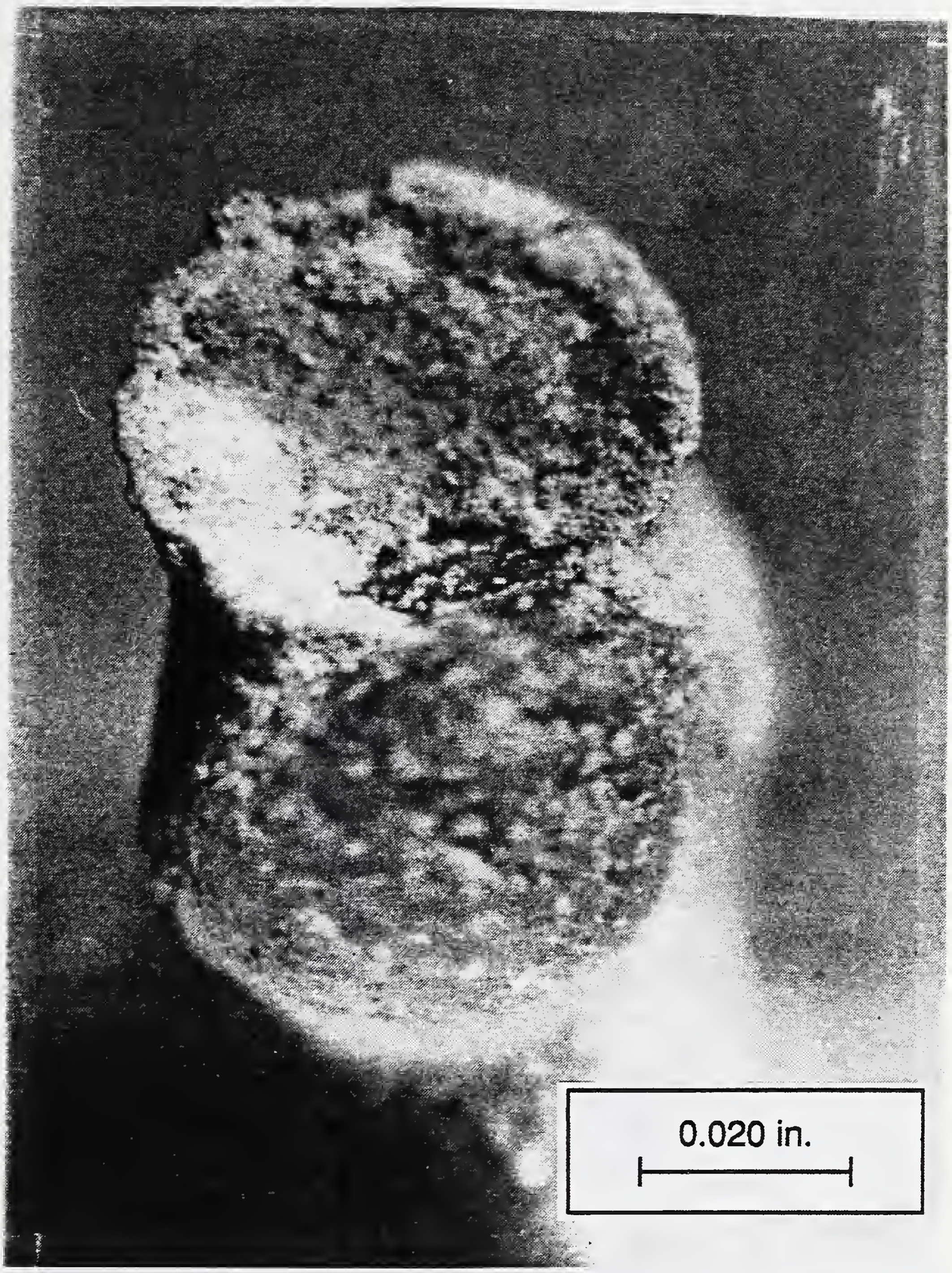

Figure 3. Specimen 1, Sand Grain Adhered to Lower Wire, 45° Viewing Angle (Scale is Approximate).

Figure 4. Specimen 1, Sand Grain Adhered to Lower Wire, 0°-Viewing Angle.

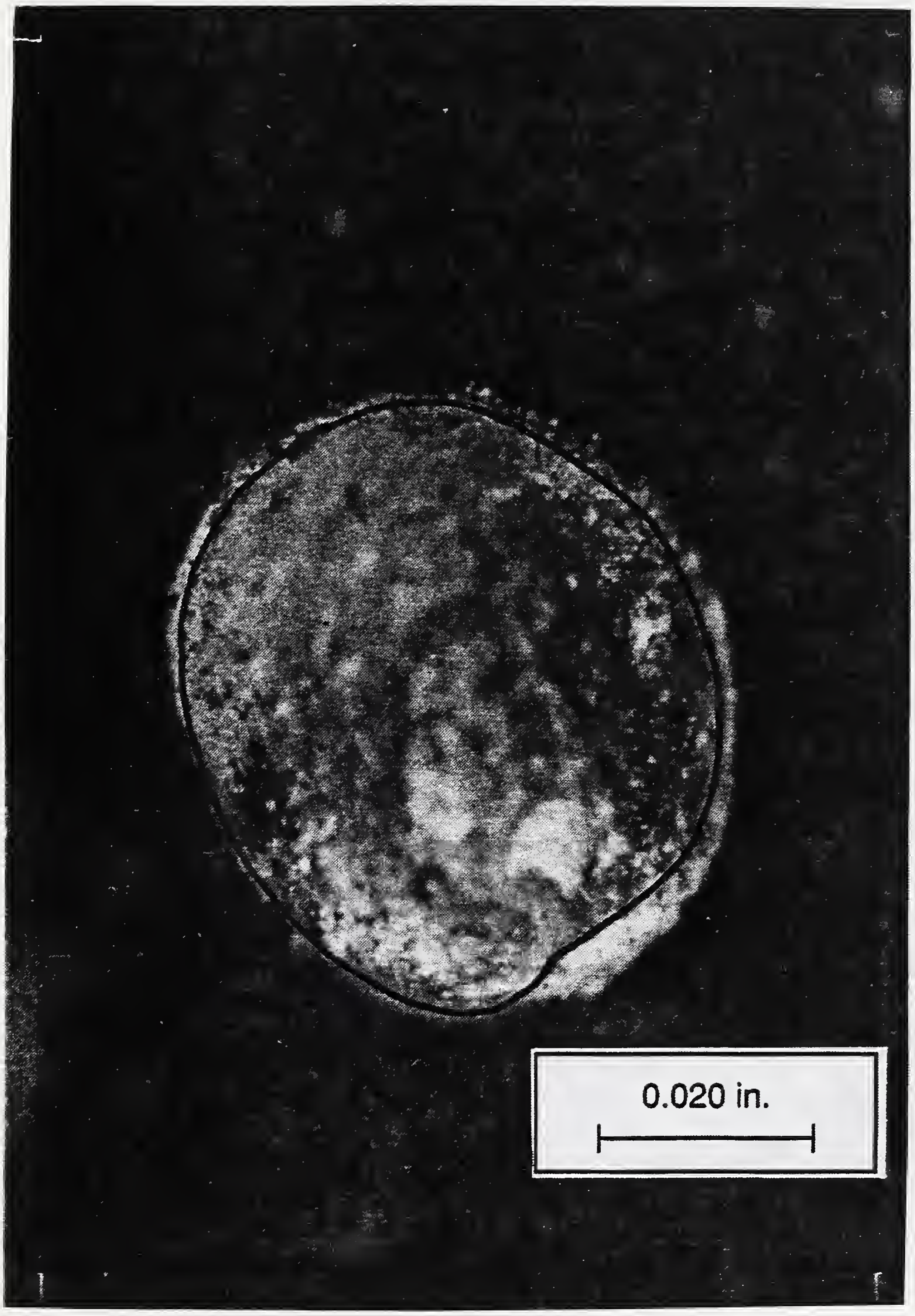

Figure 5. Specimen 1, Sand Grain Adhered to Upper Wire, Estimated Planar Failure Bond Surface Area (Used in Table 1) Shown by Enclosed Area, 90°-Viewing Angle.

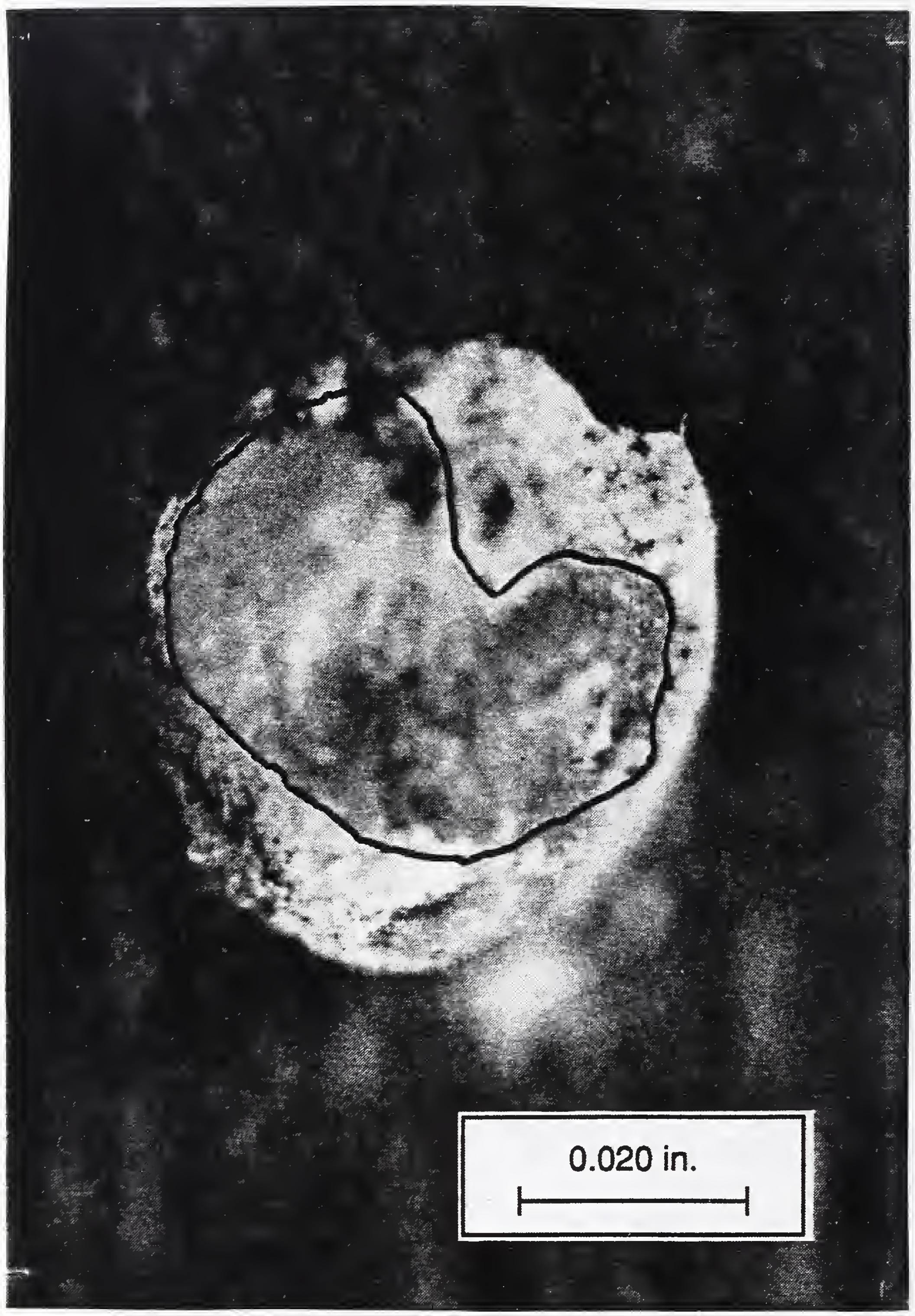

Figure 6. Specimen 2, Sand Grain Adhered to Lower Wire, Estimated Planar Bond Failure Surface Area (Used in Table 1) Shown by Enclosed Area, 90°-Viewing Angle.

Figure 7. Specimen 2, Sand Grain Adhered to Upper Wire, Estimated Planar Bond Failure Surface Area (Used in Table 1) Shown by Enclosed Area, 90°-Viewing Angle. See Footnote "d" in Table 1.

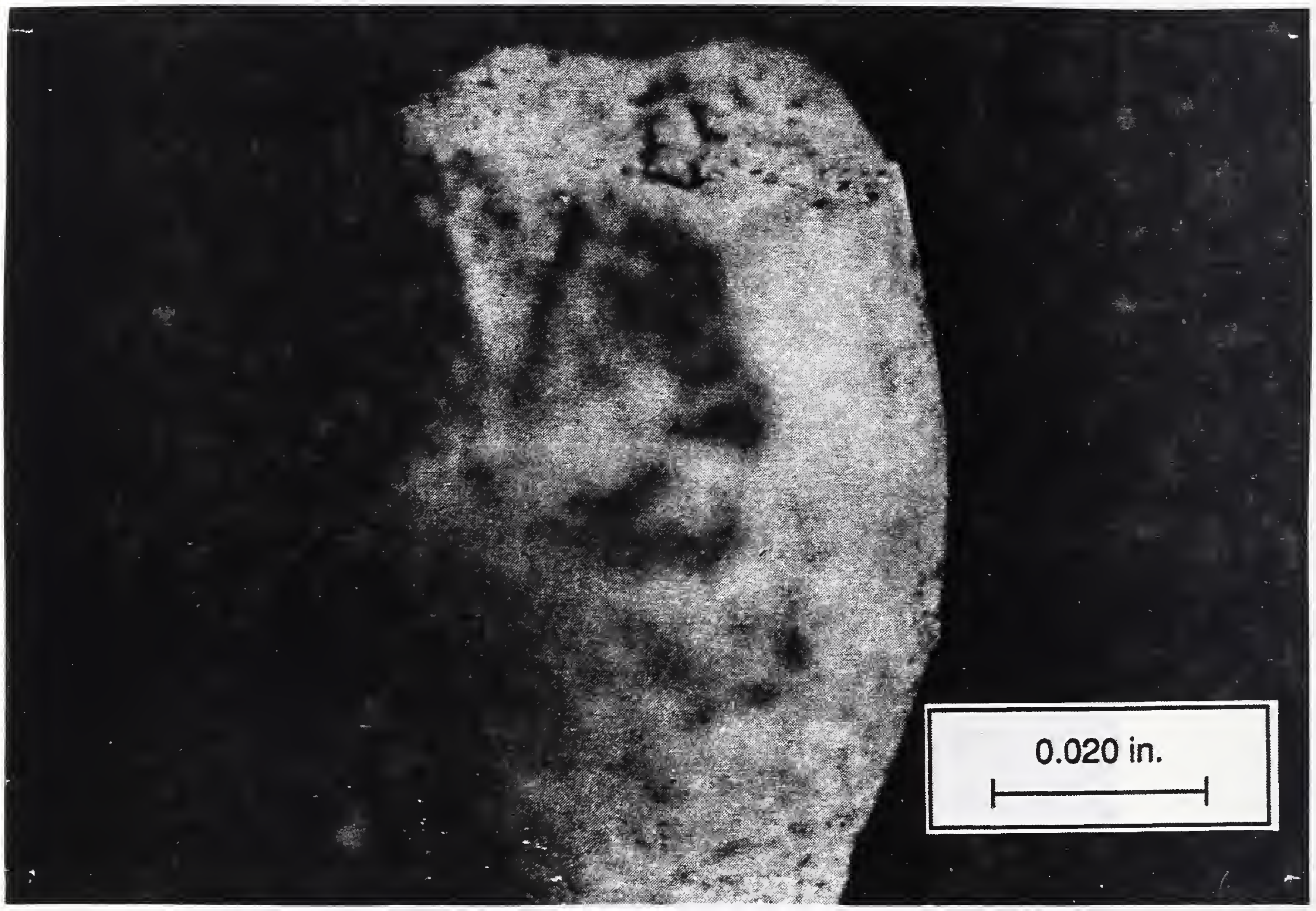

Figure 8. Specimen 2, Sand Grains Adhered to Upper Wire, 0°-Viewing Angle.

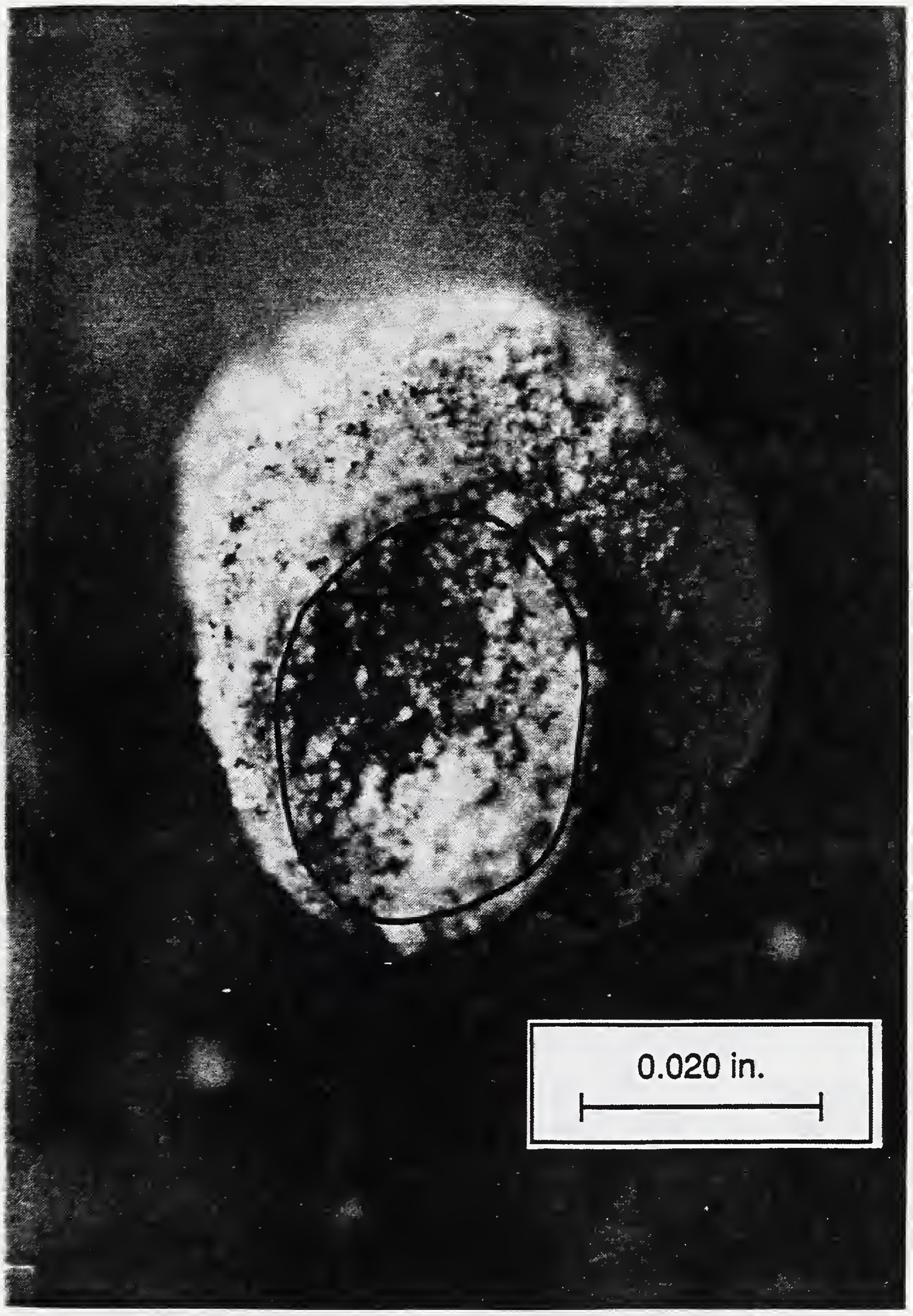

Figure 9. Specimen 3, Sand Grain Adhered to Lower Wire, Estimated Planar Bond Failure Surface Area (Used in Table 1) Shown by Enclosed Area, 90°-Viewing Angle.

Figure 10. Specimen 3, Sand Grain Adhered to Lower Wire, 45°-Viewing Angle.

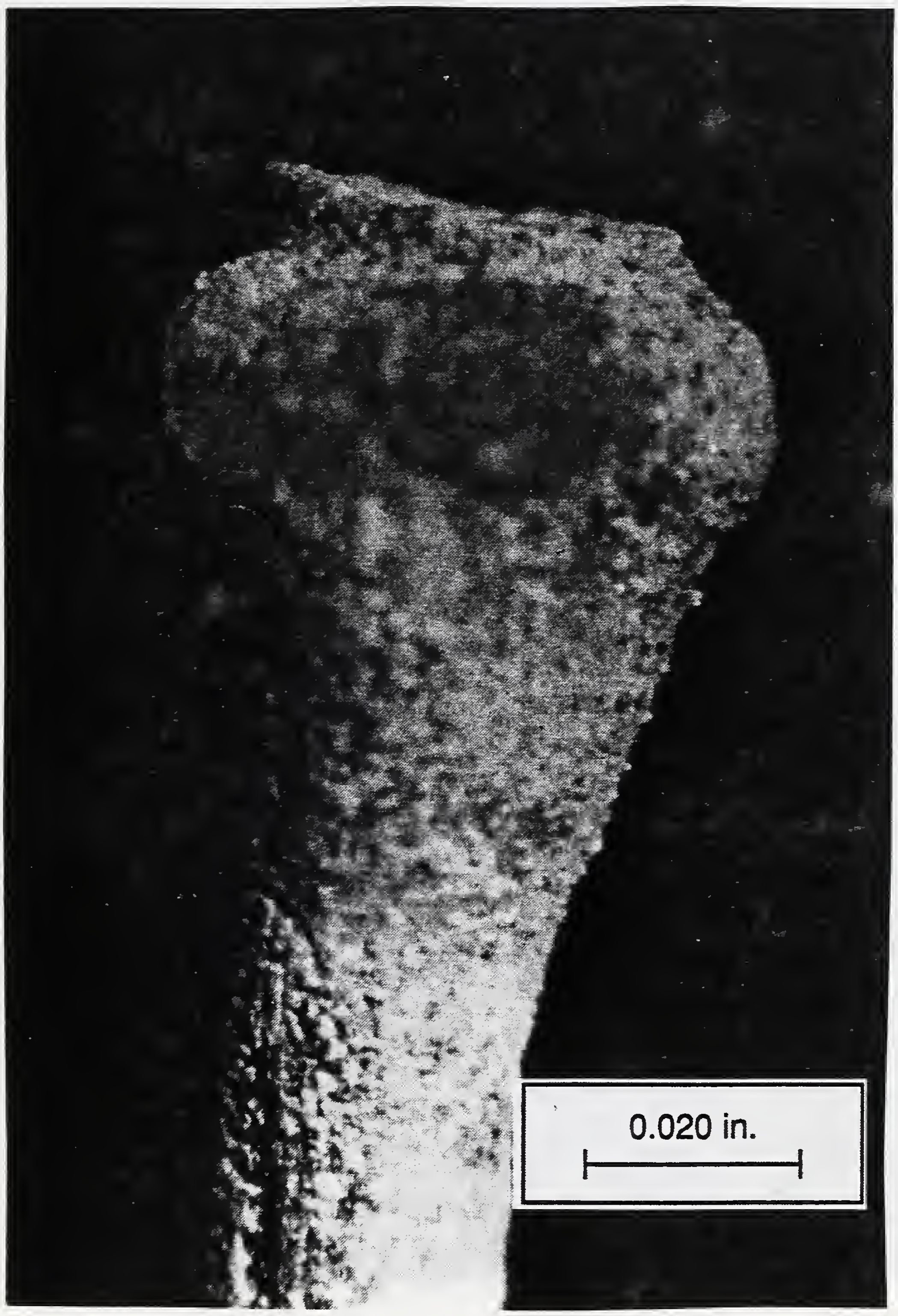

Figure 11. Specimen 3, Sand Grain Adhered to Lower Wire, 0°-Viewing Angle.

Figure 12. Specimen 3, Sand Grain Adhered to Upper Wire, Estimated Planar Bond Failure Surface Area (Used in Table 1) Shown by Enclosed Area, 90°-Viewing Angle.

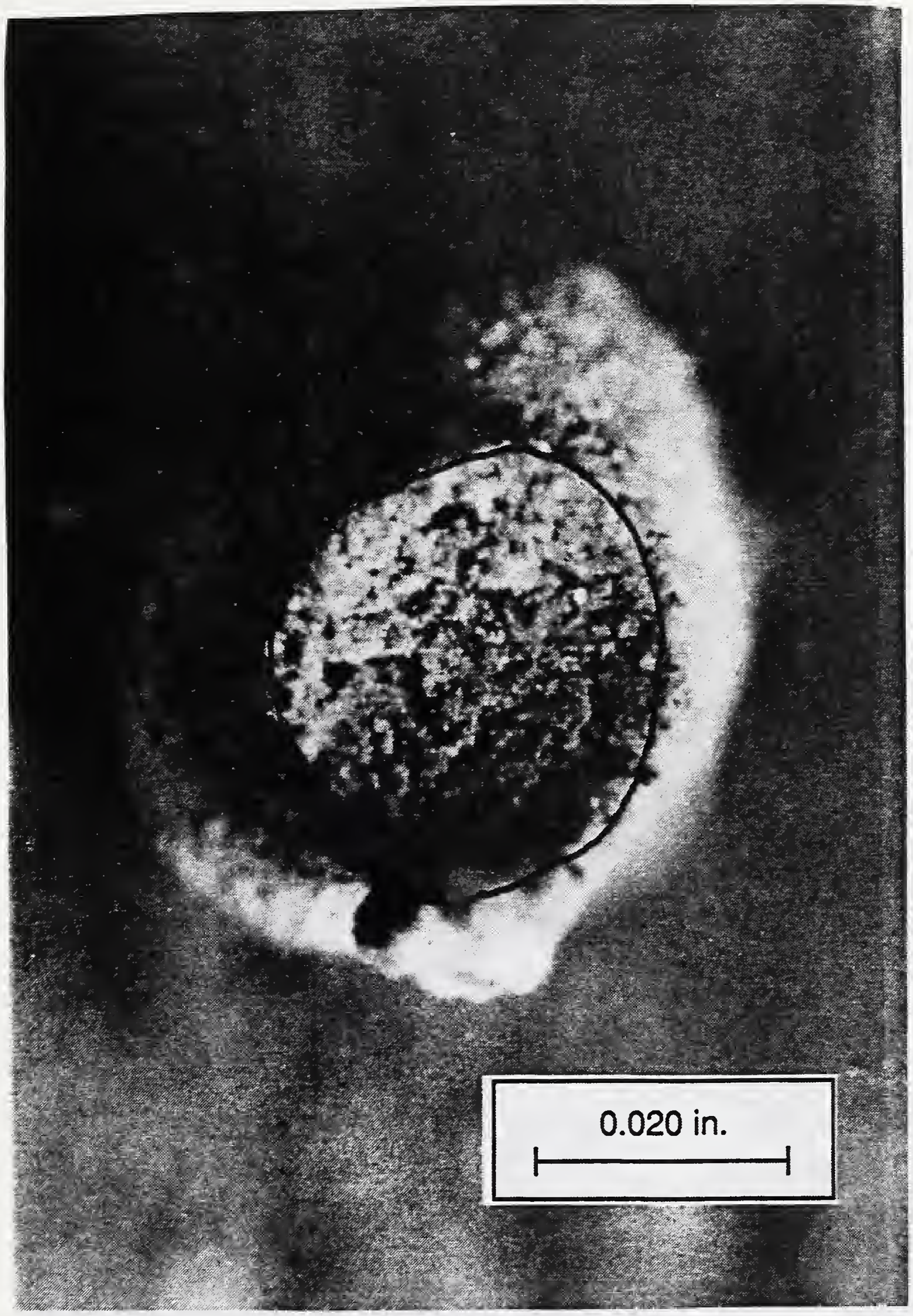

Figure 13. Specimen 4, Sand Grain Adhered to Lower Wire, Estimated Planar Bond Failure Surface Area (Used in Table 1) Shown by Enclosed Area, 90°-Viewing Angle.

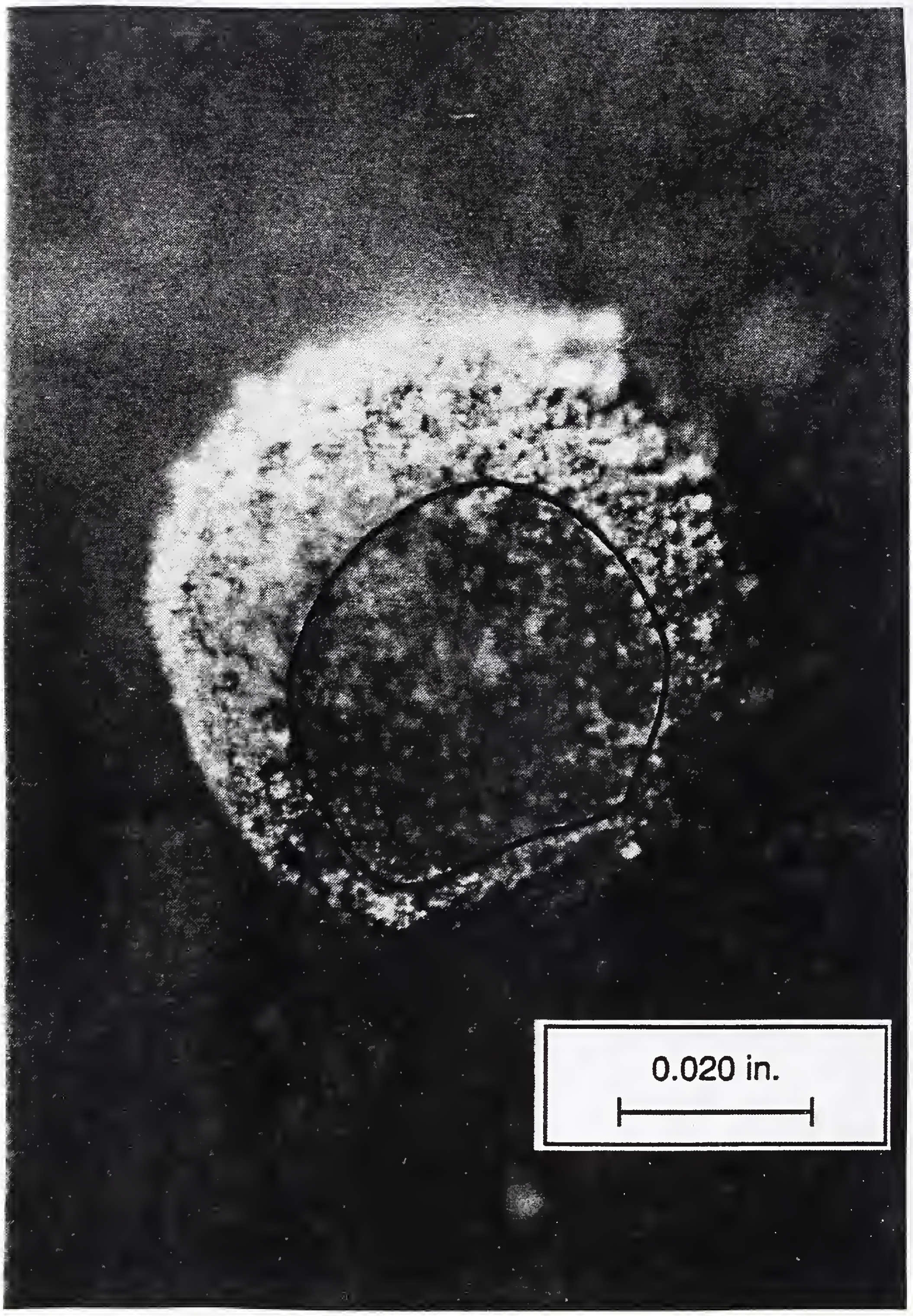

Figure 14. Specimen 4, Sand Grain Adhered to Upper Wire, Estimated Planar Bond Failure Surface Area (Used in Table 1) Shown by Enclosed Area, 90°-Viewing Angle.

Figure 15. Specimen 5, Sand Grain Adhered to Lower Wire, Estimated Planar Bond Failure Surface Area (Used in Table 1) Shown by Enclosed Area, 90°-Viewing Angle.

Figure 16. Specimen 5, Sand Grain Adhered to Lower Wire, 45°-Viewing Angle. (Scale is Approximate).

Figure 17. Specimen 5, Sand Grain Adhered to Lower Wire, 0°-Viewing Angle.

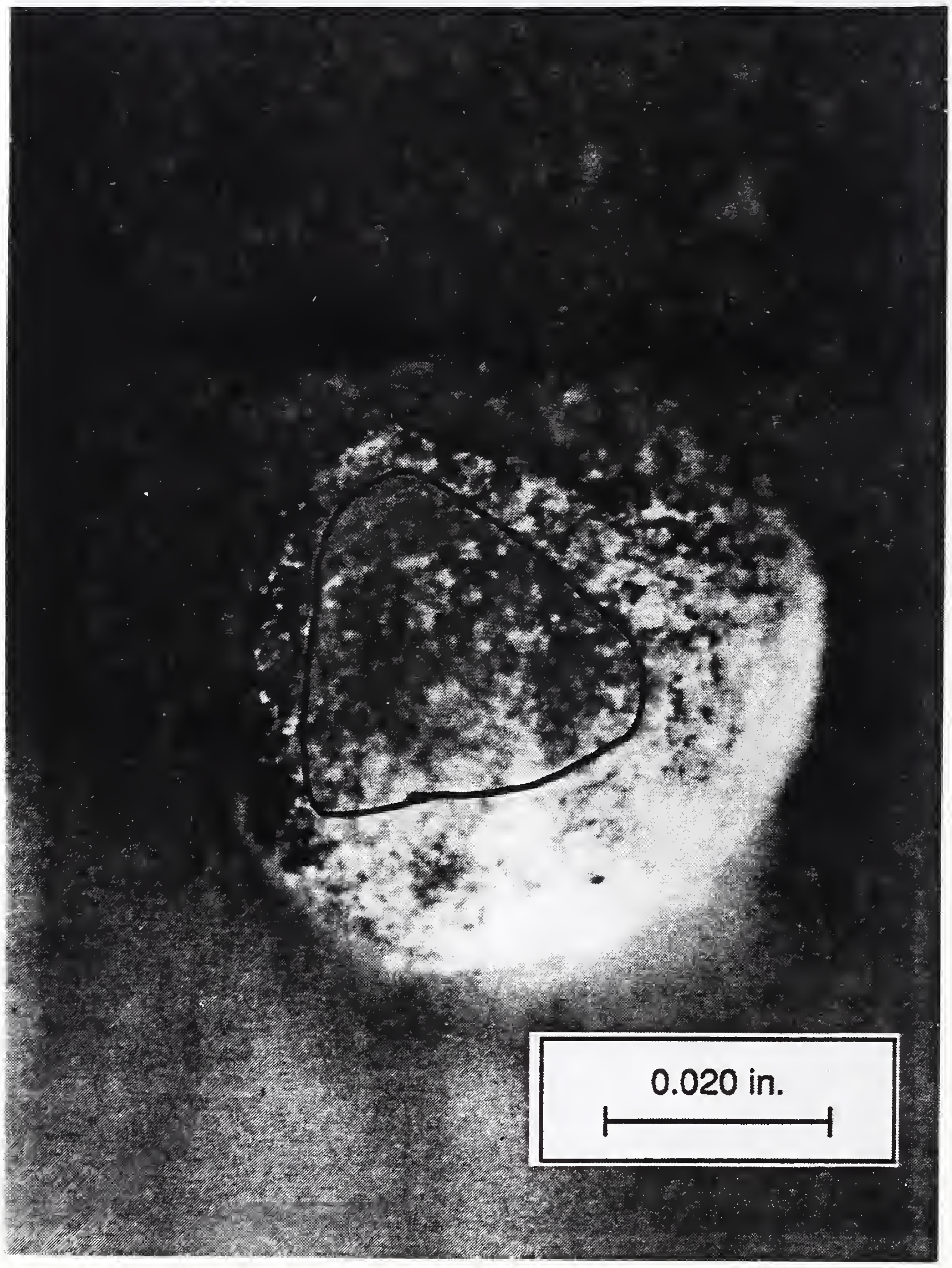

Figure 18. Specimen 5, Sand Grain Adhered to Upper Wire, Estimated Planar Bond Failure Surface Area (Used in Table 1) Shown by Enclosed Area, 90°-Viewing Angle.

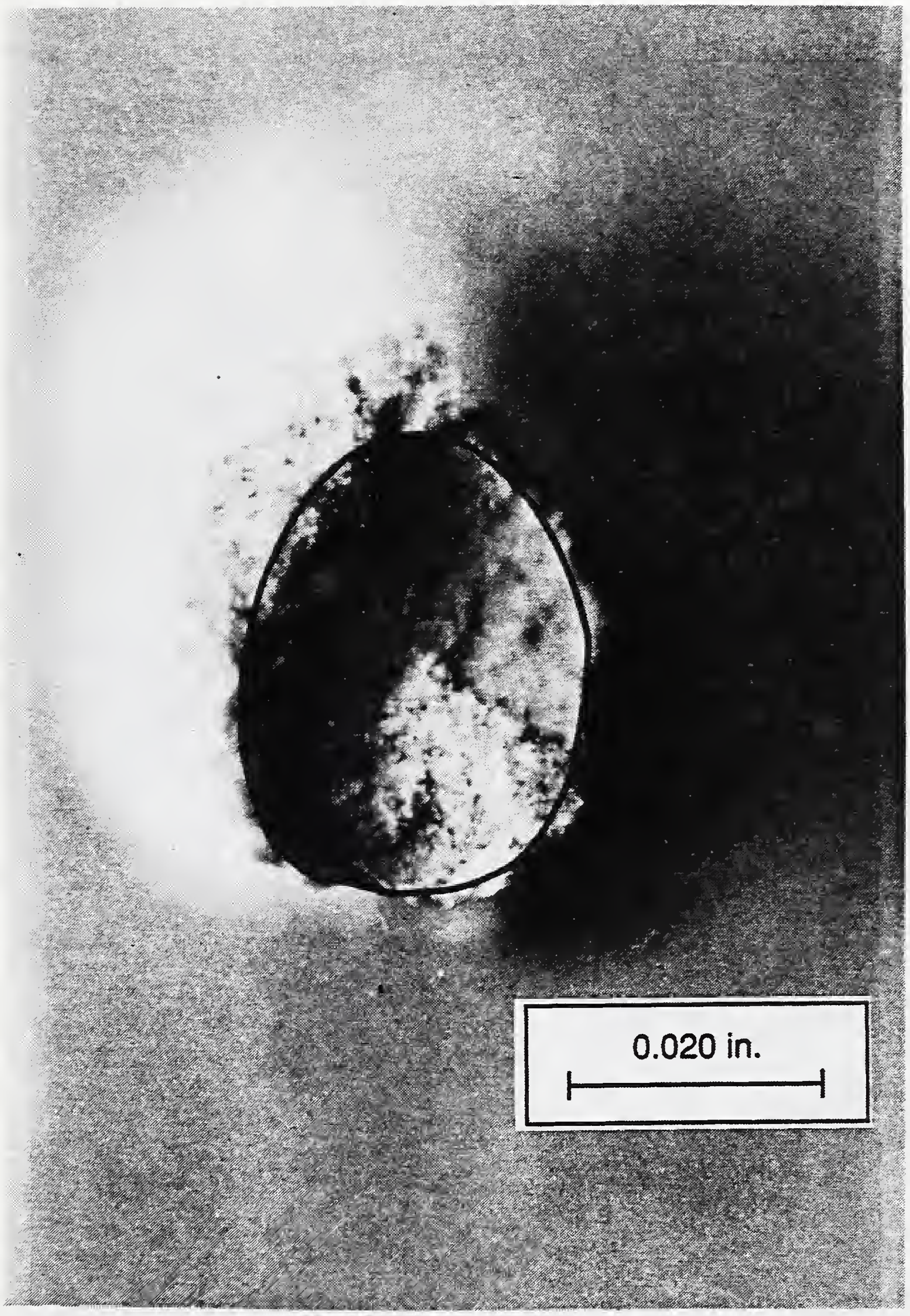

Figure 19. Specimen 6, Sand Grain Adhered to Lower Wire, Estimated Planar Bond Failure Surface Area (Used in Table 1) Shown by enclosed Area, 90°-Viewing Angle.

Figure 20. Specimen 6, Sand Grain Adhered to Lower Wire, 0°-Viewing Angle.

Figure 21 Specimen 6, Sand Grain Adhered to Upper Wire, Estimated Planar Bond Failure Surface Area (Used in Table 1) Shown by Enclosed Area, 90°-Viewing Angle.

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10. SUPPLEMENTARY NOTES

☐ Document describes a computer program; SF-185, FIPS Software Summary, is attached.

11. ABSTRACT *(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)*

A method to measure the tensile bond strength between two weakly-cemented sand grains was developed. Special microloading testing equipment was developed to measure the force required to pull apart two sand grains. To illustrate the method, bond strengths were measured in tension for six pairs of cemented sand grains. A wide range in the bond failure stress occurred and was attributed primarily to (a) difficulties in identifying and measuring the actual bond failure surface area and, (b) eccentricity in the specimens during loading. The method developed is seen as a starting point and can be used as a basis for further development. Improved techniques need to be developed to identify and measure the actual bond failure surface area and to reduce, or at least measure and account for, the eccentricity introduced.

12. KEY WORDS *(Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)*

bond; sand grains; strength; tension; test method, weakly cemented.

13. AVAILABILITY

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