

*V. R. Matricardi,¹ Ph.D.; M. S. Clark,¹ M.S.; and
F. S. DeRonja,¹ M.S.*

The Comparison of Broken Surfaces: A Scanning Electron Microscopic Study

The comparison of two broken surfaces which have the possibility of being complementary is a frequent task in forensic laboratories. Many types of materials may be examined for this purpose, including broken metal, plastic, glass, ceramics, wood, bone, and teeth.

Light microscopy has been successfully utilized in these comparisons, in particular on materials which are "brittle" and which permit the surface contours of the fracture to be easily matched. In those situations where the material fails in a ductile manner (that is, the fracture or breaking of the material is preceded by substantial deformation of the material), the fracture surfaces are severely distorted and irregular and cannot ordinarily be matched by physically placing the broken ends together.

The scanning electron microscope (SEM) can extend the range of size and roughness of fracture surfaces which can be examined because of its high resolution and great depth of field capabilities. With this instrument the size of the detail can be as small as $0.02 \mu\text{m}$ (8×10^{-7} in.) and the roughness of the specimen's surface features can be 300 times greater than that which can be examined with an optical microscope at comparable magnifications. In addition, the SEM may be equipped with X-ray spectrometers which allow the elemental composition of the specimen to be determined (at least qualitatively). Comparisons of fracture surfaces can thus be carried out both by observation of surface characteristics and by nondestructive elemental X-ray analyses.

A number of works have been published on the study of fractures using the SEM and a new field of research, electron microfractography, has been started [1,2]. Recently, Meyn and Beachem [3] used the SEM and the transmission electron microscope to match complementary fracture surfaces for the analysis of the fracture mechanism.

The SEM is used in the present study to examine the fracture surfaces of broken and cut metal wires in order to explore the possibility of rematching the separated pieces.

Experimental Methods

The wire specimens, before being examined with the SEM, are first viewed with a light microscope. A low-power stereo microscope is suitable for initial observation because of its long working distance and its three-dimensional viewing. The diameter,

Received for publication 11 Oct. 1974; accepted for publication 1 Nov. 1974.

¹Research Physicist in the Biological Science Research Unit, Chief of the Microscopic Analysis Unit, and Chief of the Metallurgy Unit, respectively, FBI Laboratory, Washington, D.C.

surface texture, and color of the wires are noted in conjunction with deformation and/or fabrication marks appearing on the surface. The length and relative position of each strand is also recorded when dealing with multistranded wires. If a higher resolution light image is required, a high quality microscope with vertical illumination can be used.

The SEM used in this study was an Etec (Autoscan® R1) having both an energy dispersive X-ray spectrometer (Kevex 5000A) and a wavelength dispersive spectrometer (Etec Autospec®).

The preparation of the wire specimens is kept as simple as possible to minimize any induced alterations. In the case of a single strand of conductive wire, the wire is clamped to a specimen stub. For this purpose a simple spring-loaded specimen stub (Fig. 1a) was constructed to allow the ruptured ends of both halves of the broken wire

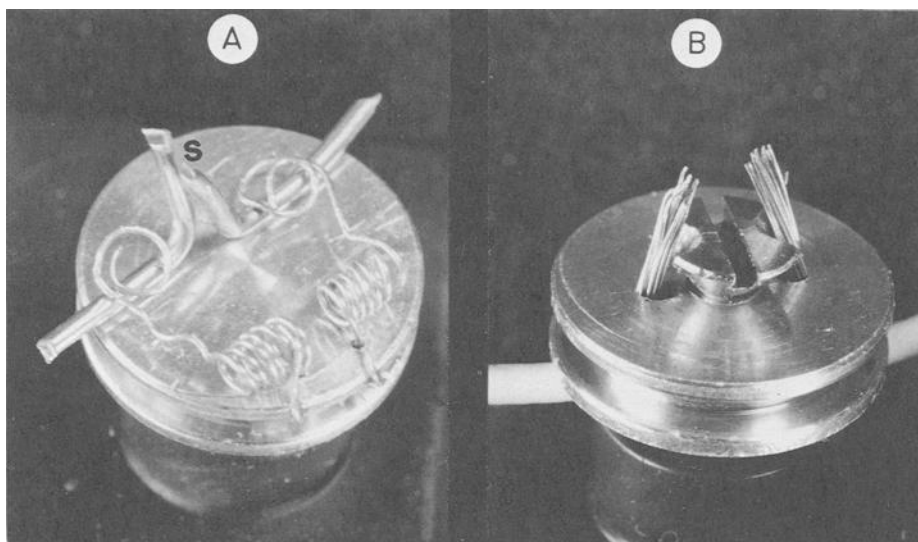


FIG. 1—(a) Specimen stub modified with two aluminum spring clips to retain both halves of a wire specimen and (b) holder used to study the break surfaces of both halves of a multistranded wire.

to be exposed simultaneously. The clamping springs are made of aluminum wire to prevent any extraneous X-rays, other than those of aluminum, from being produced. For multistranded wire a different holder was constructed, as shown in Fig. 1b. If the wire has adherent insulation, it is clamped in the holder and the central conductors are grounded by the use of miniature alligator clips. During the examination the beam is allowed to be incident only on the conductors. If the insulating layer must also be examined with the SEM, the accelerating voltage is decreased from the 20-kv normal working voltages to the 1 to 5-kv range in order to reduce charging effects. Evaporation of a metallic layer on the insulator is used only as a last resort.

In the case of multistranded wires, the strands may have to be separated from each other to provide access to the shorter strands for possible examinations. A tooth-pick or splinter rather than a pin or knife point should be used to separate the strands. Stereo pairs of both halves of the wire are taken prior to strand separation in order

to provide a measure of relative lengths and orientation of the individual strands. This information becomes useful in the subsequent attempts at matching.

The procedure used to examine two halves (A and B) of a single broken wire is as follows. Both A and B are mounted on a stub and inserted into the specimen chamber of the SEM. The specimen is lowered toward the bottom of the Z-axis to provide the greatest possible depth of field. The translation, rotation, and tilt controls are maneuvered until a normal view of the break surface of A is obtained. Various micrographs at different useful magnifications ($\times 100$ to $\times 10,000$) are obtained after proper focusing and cycling of lens current to remove hysteresis effects. Fracture Surface B is then brought into the field of view, aligned, and brought into focus by adjusting the Z-axis position control. Consequently, the objective lens current can be held fixed during this process thus maintaining a fixed total magnification. A sequence of micrographs is taken of Surface B with magnifications matching those taken of Surface A.

For matching ends of broken wires, the greatest useful detail is obtained when the fracture surface is viewed along its perpendicular. If the two fracture surfaces are viewed at two different angles a foreshortening will occur, making the subsequent matching process difficult, if not impossible, to perform. If the fracture surface is viewed from an angle, then this angle must be exactly duplicated when viewing the complementary fracture surface.

The micrographs of Surfaces A and B (Fig. 2) were taken on Polaroid 55 P/N films. The prints are mirror images of each other and this required that they be placed face to face before matching of distinguishing features could be achieved, an awkward process.

A simpler way to make the comparison is for the negative from one of the micrographs to be used to make a reverse print. This print can then be used for a side-by-side comparison with a micrograph print from the other surface. This method was employed to prepare the micrographs which appear later in this paper.

One can also work directly with the photographic negatives. The two negatives can be superimposed on a light box (back to back) and compared in that manner. Transparencies from the two surfaces may also be colored differently and the superposition of the two will show areas of correspondence and disparity by the appearance of the transmitted spectrum. The transmitted light may be joined to a filtered photomultiplier and the percentage area of correspondence between two surfaces may be calculated automatically.

Another method for comparison which was recently installed is to obtain electronically a mirror image by reversing the scan direction. This technique provides two positives which are not mirror images of each other. Another option was added by recording the image of A electronically in a special video recorder (Etec Vistascan[®]) and placing the electronically inverted image of B alongside the first. The alignment can now be carried out with greater ease, having the two images (from Surfaces A and B) side by side at identical magnifications. Two slide projectors may also be used for comparison purposes with alternating light shutters. On the viewing screen the two images are projected alternately, the magnifications and orientation adjusted, and the contours compared.

The matching of fracture surface features is best achieved if small areas of the micrographs are considered individually. As stated above, small variations in the viewing angle will cause foreshortening, which becomes more evident as the area being viewed becomes larger.

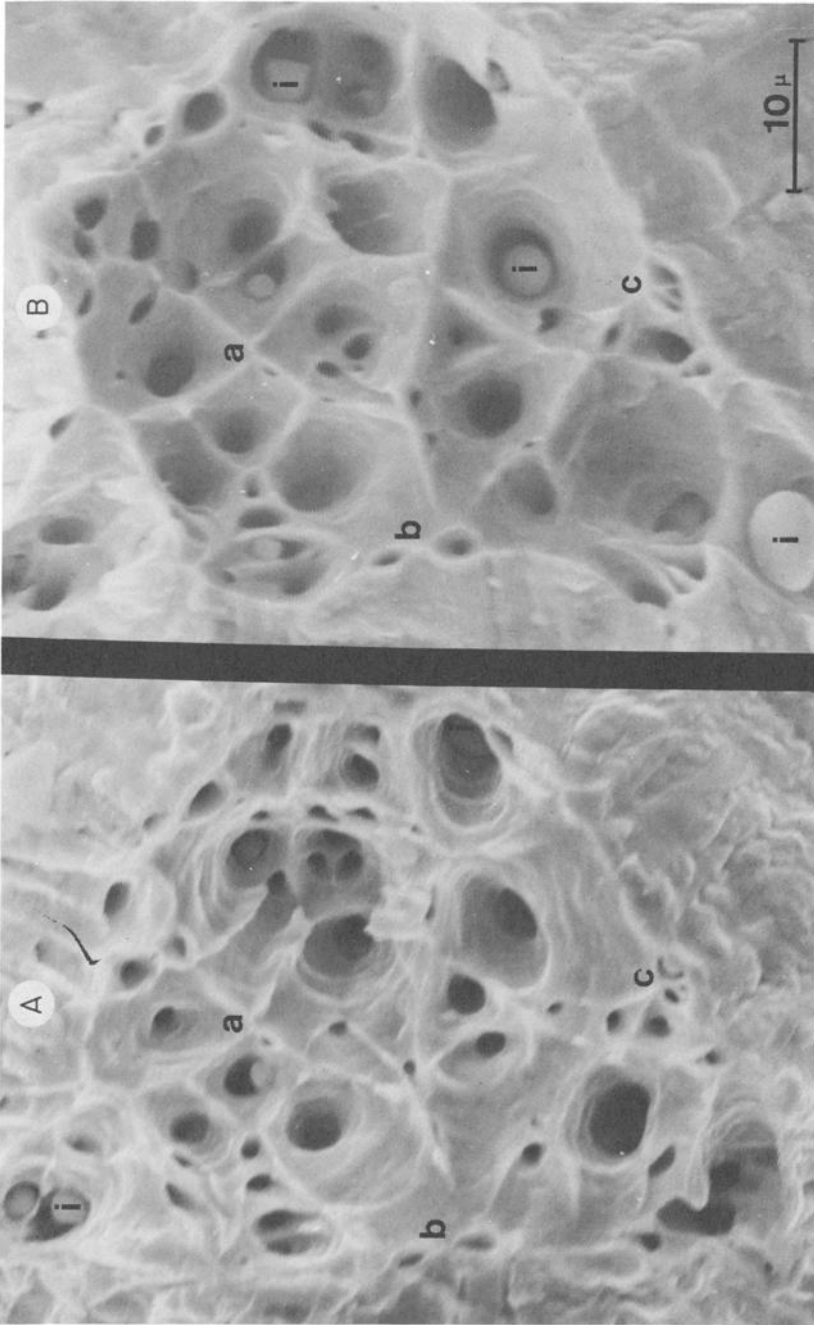


FIG. 2—Matching ends of a silver-coated copper wire broken in tension. The break surfaces are viewed along the longitudinal axis of the wire with letters a, b, and c designating some points of correspondence. Inclusions (designated by the letter i) around which some of the voids had formed have no corresponding structure appearing on the mating break surface (20-kV accelerating potential).

Comparisons of the images obtained by deflection modulation were also attempted but provided no significant advantages over the normal (intensity modulated) images.

In most cases where matching is attempted, the energy dispersive analyzer (XES) is also used. A small area of the specimen (with the flattest surface topography) is chosen and an X-ray spectrum is obtained. The X-ray spectrum may be plotted by a Hewlett-Packard X-Y plotter for later comparison, or it may be stored in the electronic memory of the KeveX unit and compared directly on the video monitor. Qualitative results are usually obtained within a period of one to two minutes. The resolution of the peaks of this type of analysis is roughly 160 eV.

If quantitative results or higher resolutions are required, the wavelength spectrometer is used. Four possible analyzing crystals are available to choose from, depending on the wavelength of the characteristic X-rays. The time required to achieve a full analysis is, however, much longer than that obtainable by XES (on the order of one half hour).

The authors have examined over 80 wire pairs broken under various controlled conditions, as well as wires from actual criminal cases. The wire specimens examined were separated by tension, shear, and torsion forces. In addition, some wire specimens were severed by diagonal cutters and by sawing.

Results

Wire Broken in Tension

Figure 2 shows electron micrographs of the fracture surfaces of a 122- μm (0.0048-in.) diameter, silver-coated copper wire, broken in tension and viewed along the axis of the wire. The micrographs show a pattern of voids and ridges which were formed during the early stages of metal deformation. The fracture surfaces have a diameter of about 40 μm (0.0016 in.) and both surfaces are concave. Because the entire area of the fracture surface is in focus, the surface has to be examined off axis to determine its concavity.

According to present theory of ductile fracture in metals [4], the application of stress first causes numerous small voids or cavities to be formed at various sites within the metal. As the stress increases the voids grow, coalesce, and eventually form a crack which leads to complete separation of the metal. It is the two-dimensional arrangement of the voids and the associated ridges between them which most often allows the two halves of broken metal to be matched.

The correspondence of the void arrangement can be noted between the two wire halves in Fig. 2. The letters in the micrographs designate some of the points of correspondence and may be used by the reader to prove the complementarity of the two surfaces.

Not all wire breaks will provide as complete a match as is shown in Fig. 2. Some of the reasons for this lack of correspondence are scarcity of details, contamination, break surface not being perpendicular to the wire axis, and mishandling of the fracture surface prior to examination.

Figure 3a shows two broken-stranded electrical conductors as they appeared when received at the FBI Laboratory in connection with a criminal matter. Each conductor contained 20 copper wire strands of 150- μm (0.006-in.) diameter. Preliminary optical and physical examinations revealed nothing to indicate that the broken conductors could not be complementary. Optical microscopy further revealed that all of the strands in each conductor had broken in tension, but that the fracture surfaces could not be compared by light microscopy because of depth of field limitations. Therefore, the

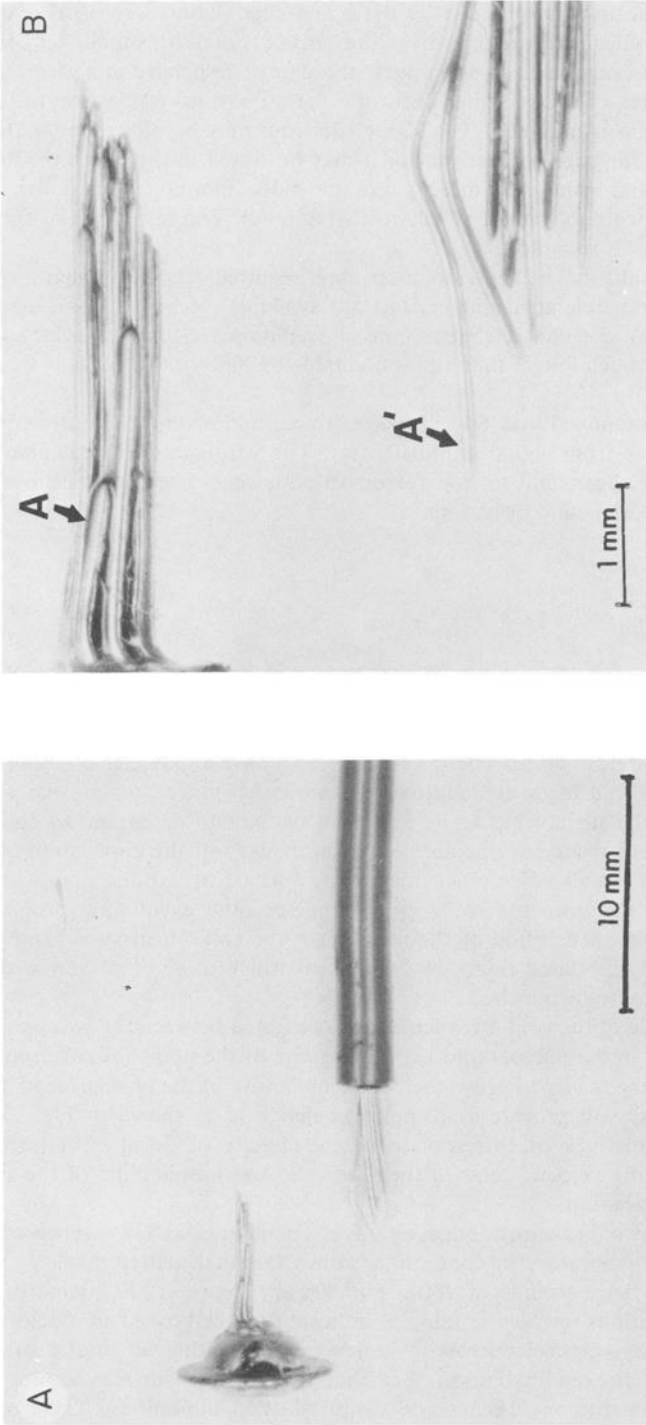


FIG. 3—(a) Broken stranded electrical conductors as received at the FBI Laboratory in connection with a criminal matter and (b) close-up view of the broken wire strands. Letters designate the wire strands chosen for SEM fracture comparisons.

longest wire strand in the one conductor was removed for an SEM comparison with the shortest wire strand in the other conductor (A and A' in Fig. 3*b*). Because of the small size of the strand pieces they could not be clamped, but were mounted on individual specimen stubs with a conductive cement.

Figures 4*a* and 4*b* show electron micrographs of the fracture surface areas for the two strands. Based on the matching characteristics exhibited, the wire strands were identified as having been broken from each other.

In a separate study, the variation of the speed at which the tensile breaks were made (20 to 75 cm/s) did not have a reproducible effect on the fracture appearance of ductile metals. Thus, a differentiation could not be made between a break in a ductile metal caused by a slow pull or one caused by a sudden jerk.

Figure 5 shows the fracture surfaces of 125- μm (0.005-in.) tungsten wire broken in tension. The tool used to pull the wire had indented the periphery slightly and may have been responsible for the radial cracks. Electron micrographs of the two complementary fracture surfaces are shown in Figs. 5*a* and 5*b*.

Wire Broken in Shear

Figures 6*a* and 6*b* show micrographs of corresponding portions of a sheared aluminum wire.

When a shearing tool severs a wire, particularly a ductile wire, it will leave tool marks over a large portion of the severed area, if not the full area. In addition, during the severing process the two surfaces may rub against each other, thereby removing detail which might be useful in matching. For these reasons the matching process for wires severed by shear forces is more difficult than for wires broken in tension.

Figures 7*a* and 7*b* show micrographs of small sheared areas on the severed ends of a steel spring wire of 350- μm (0.014-in.) diameter. The major portions of the severed ends were completely covered by tool marks from the shearing tool. It should be noted that a filament originally parallel to the wire's axis will be displaced in opposite directions on opposing halves of the wire.

Wire Broken in Torsion

The center of rotation of a 125- μm (0.005-in.) tungsten wire broken in torsion is shown in the micrographs of Figs. 8*a* and 8*b*. Although the outer areas exhibited some matching characteristics, these details are not as clear as those present near the center. The fracture surfaces for both ductile and brittle metals can be recognized as having been broken by torsion because of the very characteristic patterns apparent in the micrographs.

Wire Severed by a Diagonal Cutter

Figure 9*a* shows two halves of a steel wire having a diameter of 0.9 mm (0.035 in.) which had been cut with a diagonal cutter. In this situation, the cutting blades of the tool were traveling in the same plane (as opposed to a shearing action). The opposing blades notched the wire to a depth sufficient to produce a separation by fracturing, leaving a wedge-shaped fracture surface.

When the fracture surfaces are viewed along the wire axis with the SEM, sufficient characteristic details become visible to provide a match (see Figs. 9*b* and 9*c*).

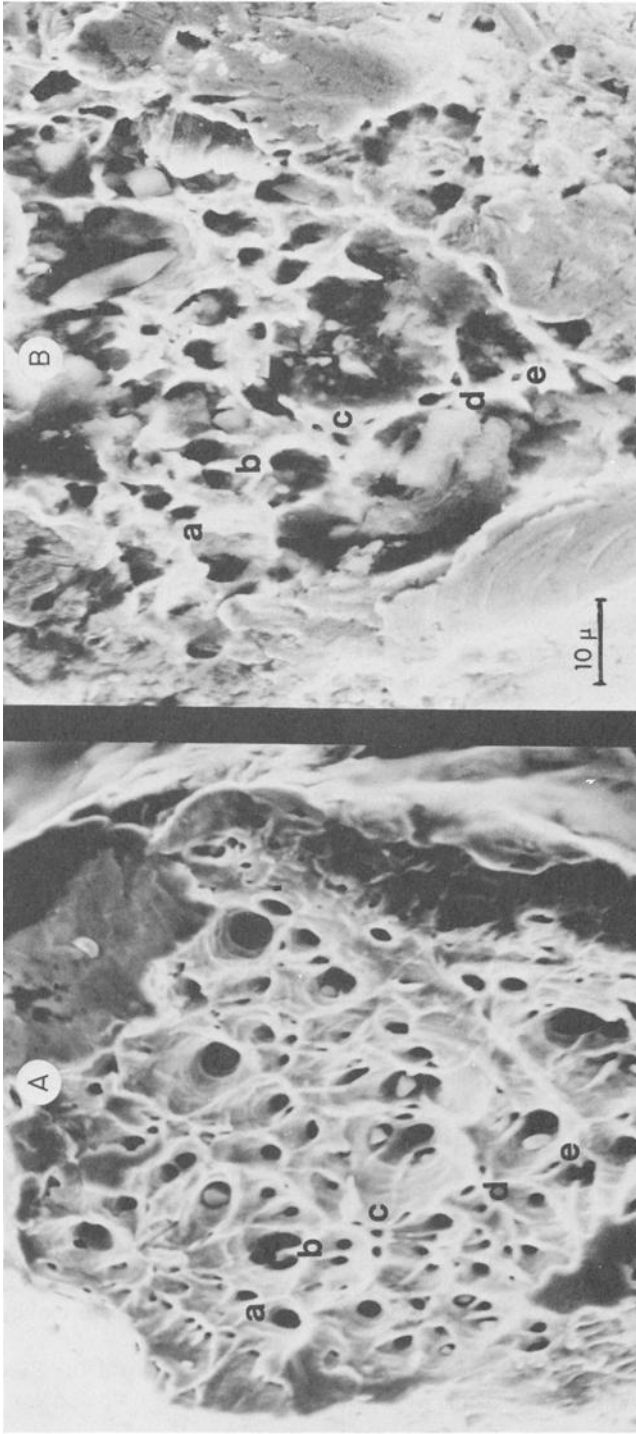


FIG. 4.—Fracture surfaces of wire strands A and A' shown in Fig. 3b. Letters designate some of the points of correspondence upon which the strands were identified as having been broken from each other. Fracture Surface A is on the left.

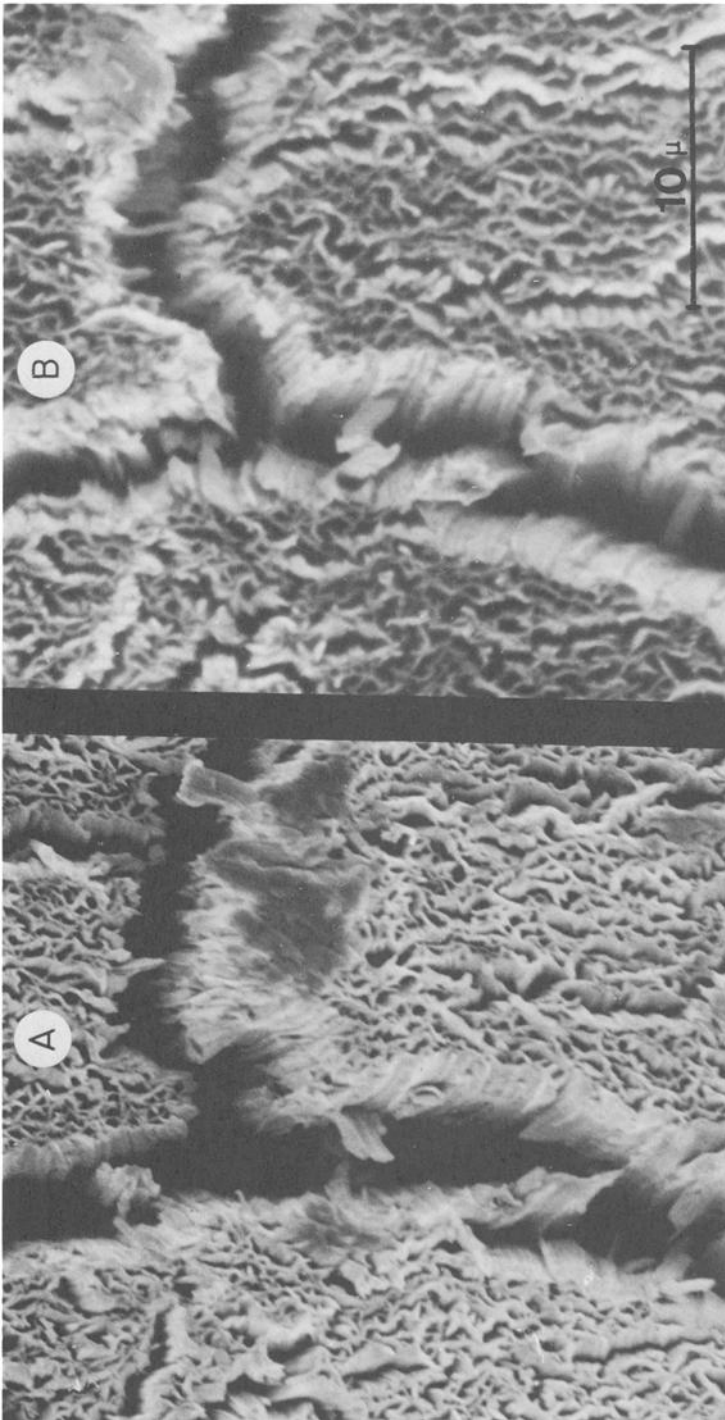


FIG. 5—Sections of the matching end of a 125- μm diameter tungsten wire broken in tension (20-kV accelerating potential).

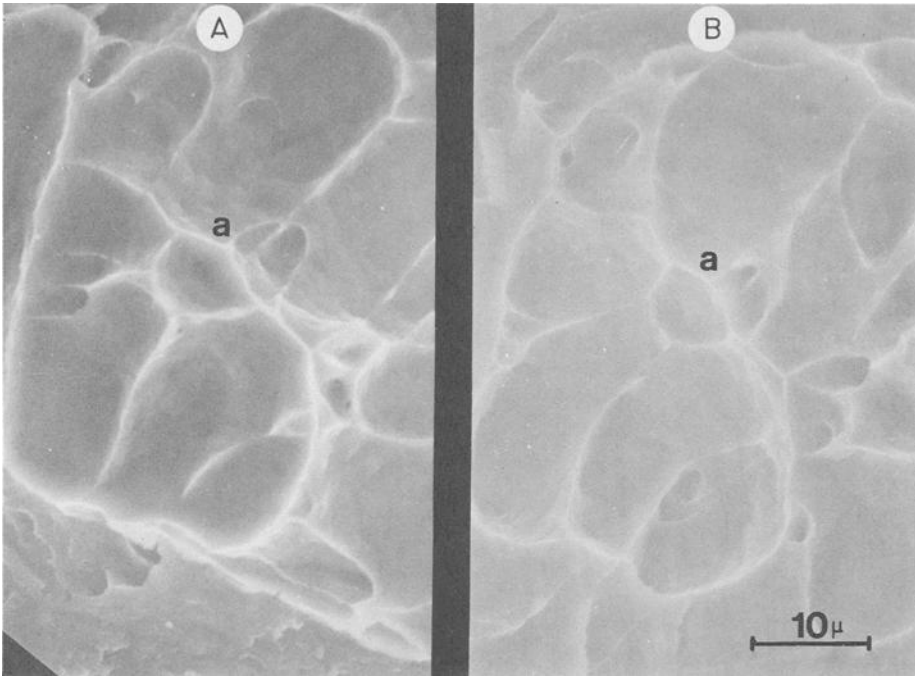


FIG. 6—Matching areas of the ends of an aluminum wire of 330- μm diameter severed in shear (20-kV accelerating potential).

Wire Severed by Sawing

Figure 10a depicts a strand from a steel wire rope cut with a saw. As is usually the case, the saw cut through an appreciable percentage of the cross-sectional area (marked by the letter S), but the final separation occurred by fracture. It should be emphasized that this fractured area is what allowed the two halves to be matched. Figures 10b and 10c are the two corresponding surfaces of the fracture taken at higher magnification.

Elemental Analysis

As a preliminary step in the comparison process, compositional characteristics of the pieces of wire involved should be determined. If substantial differences are found, further comparisons are not justified. The energy dispersive X-ray spectrometer is ideally suited for these compositional comparisons.

The simplest comparison occurs when the two broken halves are made of a homogeneous material (for example, copper wire). A more complex case would involve the comparison of a nonhomogeneous material such as a copper wire with a silver coating. Figure 2 shows the break surface of a wire on which an X-ray analysis was performed. The break surface itself provides the Spectrum A shown in Fig. 11; however, the surface of the wire has a high concentration of silver, as shown in Spectrum B. Spectrum C was obtained from the necked-down region of the wire and shows a smaller concentration of silver. Matching spectra were obtained from the other half of the wire.

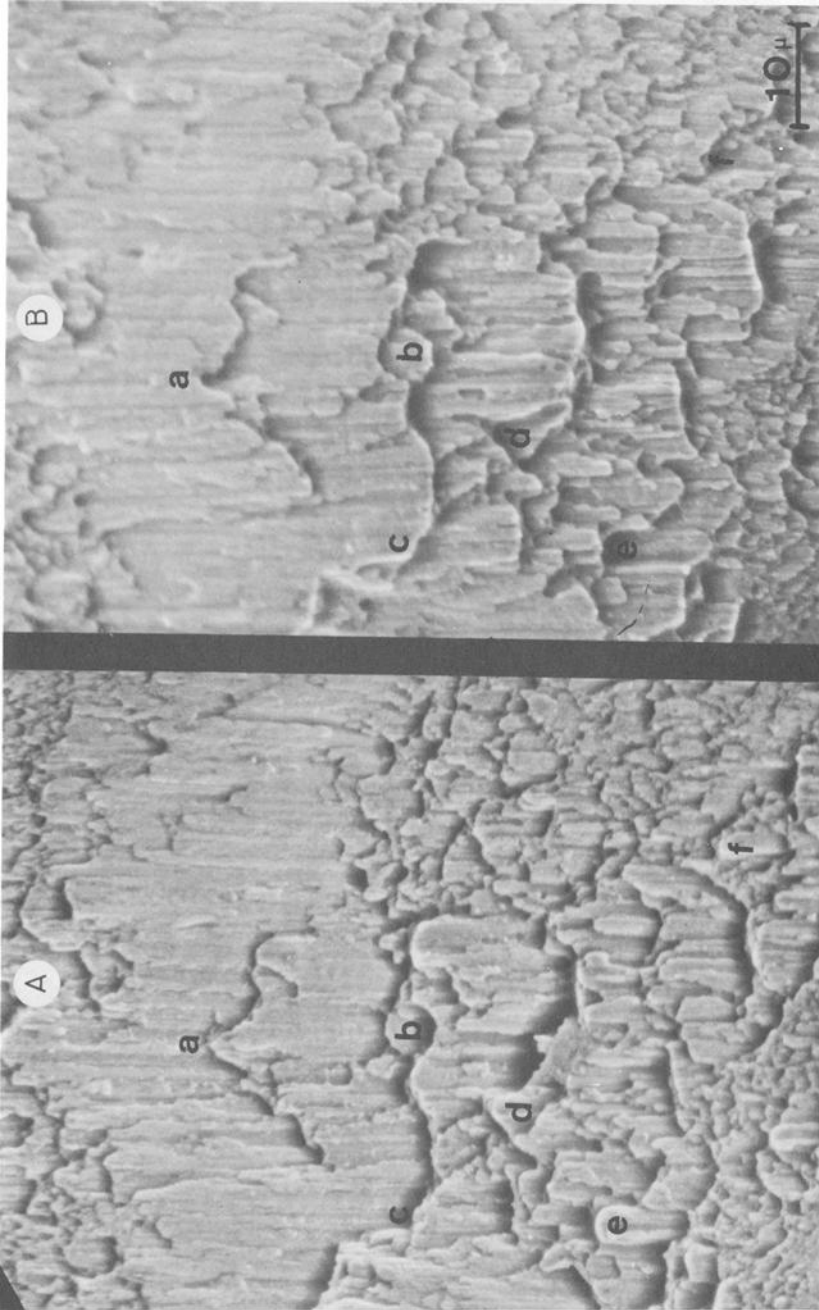


FIG. 7—Part of the fracture surface produced by shearing of a steel spring wire of 350- μm diameter. Letters a through f represent some of the areas of correspondence (20-kV accelerating potential).

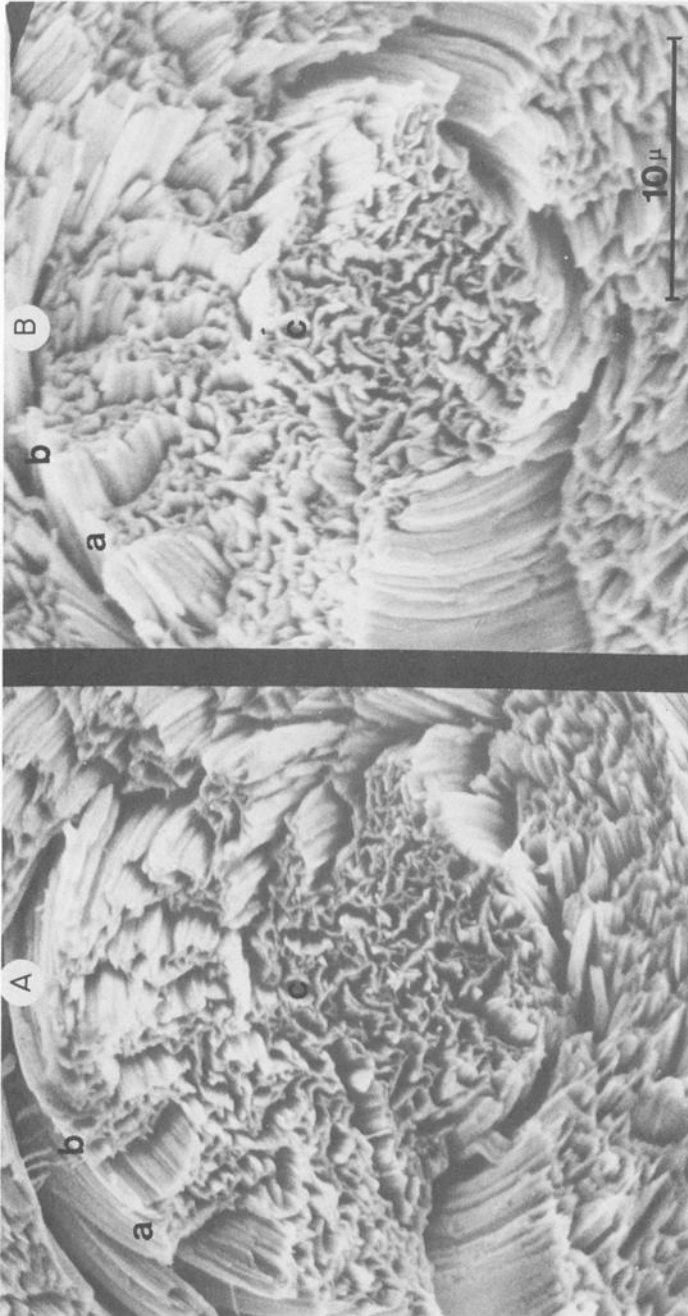


FIG. 8—Area surrounding the center of rotation of the break surface of a 125- μ m diameter tungsten wire broken in torsion (20-kV accelerating potential).

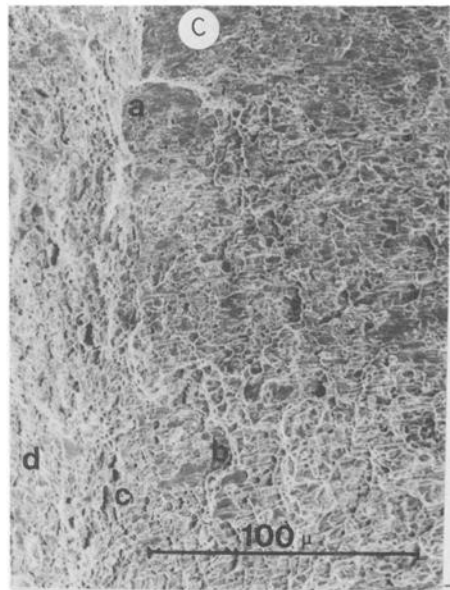
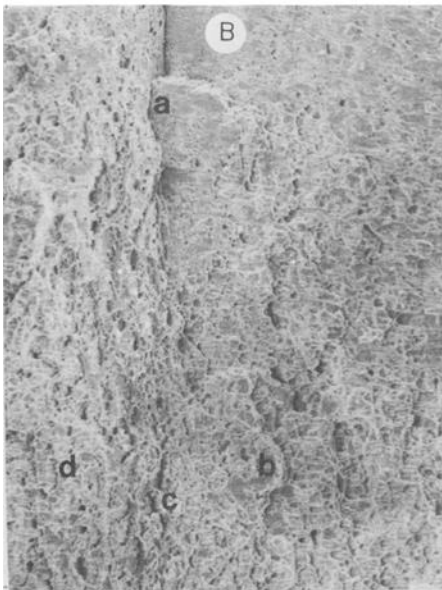
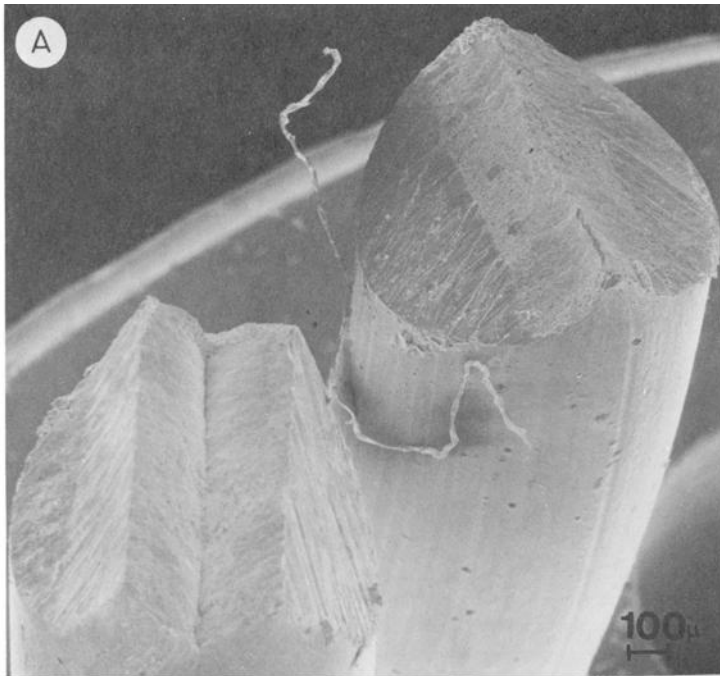


FIG. 9—(a) Steel wire of 0.9-mm diameter severed by a diagonal cutter and (b and c) corresponding fracture of the wire pieces shown in (a) but viewed along the longitudinal axis of the wire.

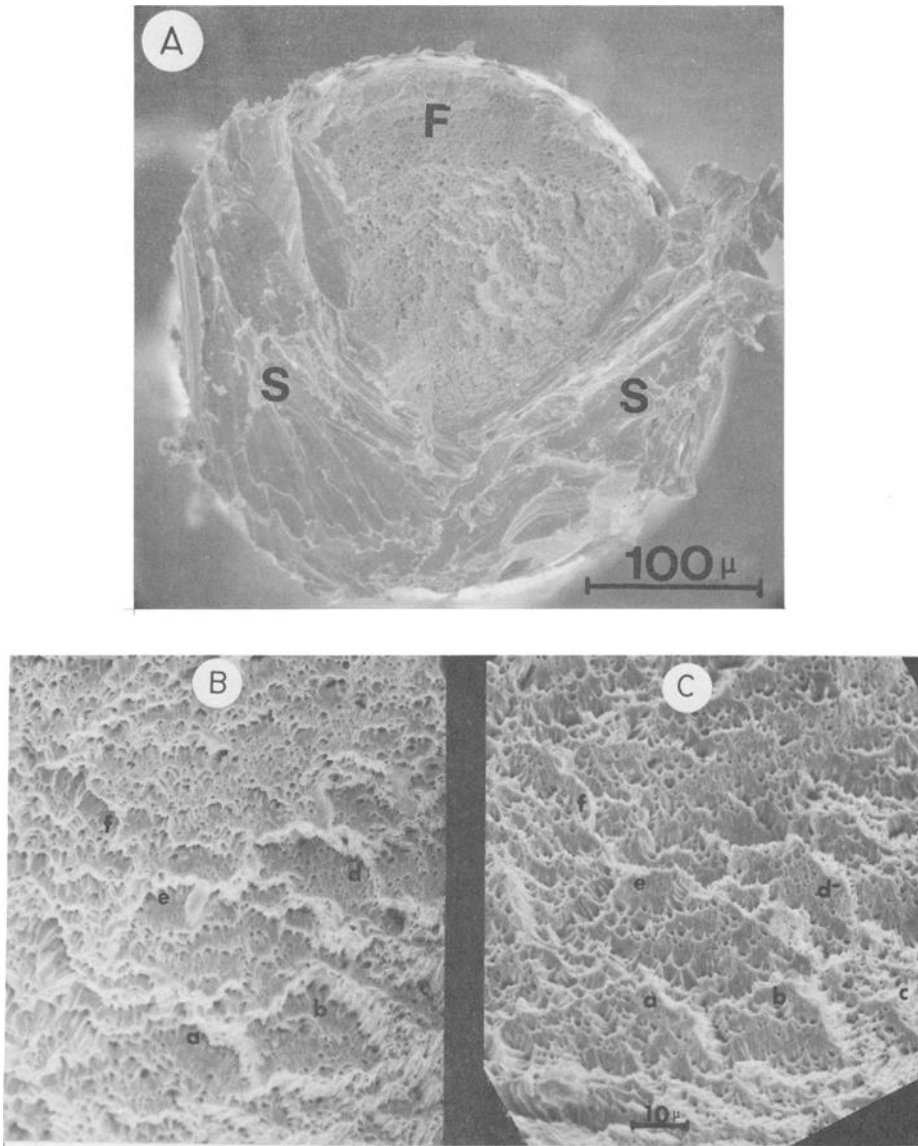


FIG. 10—(a) Strand from a steel wire rope cut with a saw. The sawing action has cut the area marked by the letter S. Final separation occurred by fracture (Area F). (b and c) Area of wire shown in (a) which had fractured and which can be used for rematching.

If correspondence of the X-ray spectra were the only results available, the conclusion could only be that the two wires have similar composition. If, however, the composition of the wire is relatively rare, the probability of the two wires matching becomes proportionately larger.

Sometimes the distribution of a particular element on the surface of a sample is

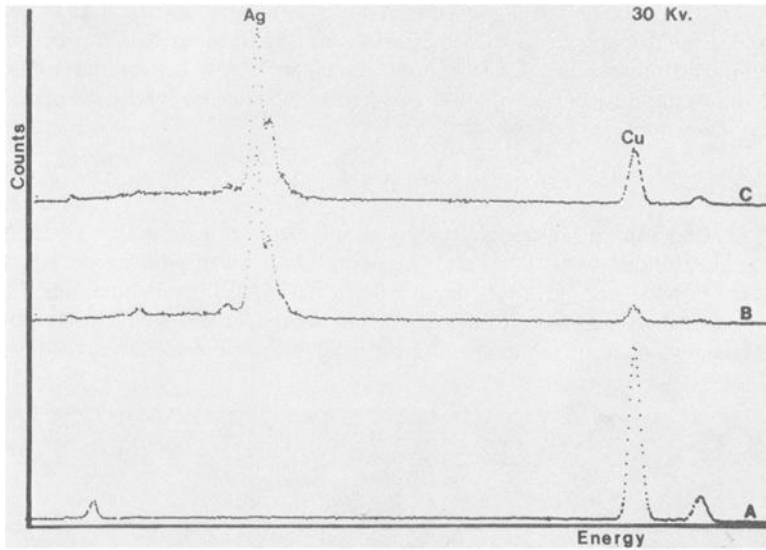


FIG. 11—X-ray spectra of the silver-coated copper wire shown in Fig. 2 obtained with the energy dispersive spectrometer. A corresponds to the break surface; B corresponds to the surface of the wire; C corresponds to the necked-down region of the tensile break.

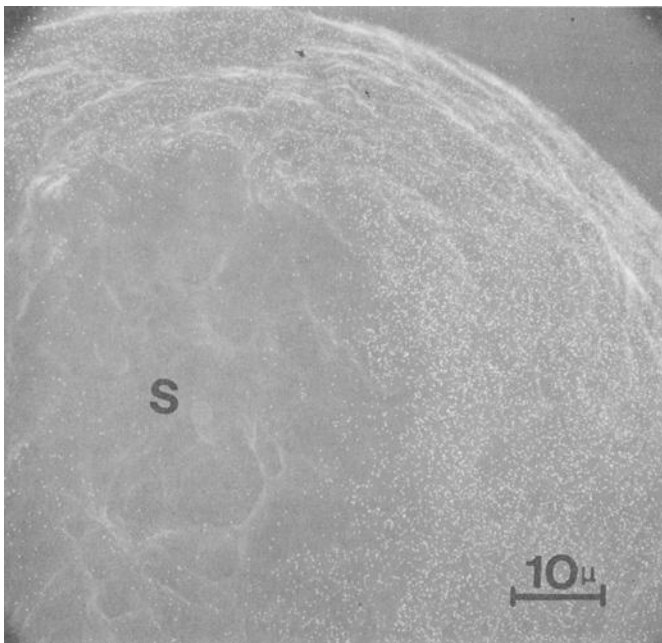


FIG. 12—Superposition of SEM image and dot pattern showing the localization of silver on a silver clad wire. Silver is not present on the break surface(s) but appears only on the surface of the wire (30-kV accelerating voltage).

characteristic. In this case the X-ray spectrometer can be focused on that particular element and a micrograph of its distribution on the specimen surface can be obtained. For a recent article presenting this technique, see the article by Brown and Johnson [5]. Figure 12 shows the distribution of silver on the silver-coated copper wire superimposed on an SEM image.

Light Microscopy

Figures 13a and 13b are, respectively, optical and electron micrographs of the ends of the same multistranded wire. The light micrograph was taken with a $\times 6.5$ (0.18 NA) objective to provide the greatest depth of field. If a higher numerical aperture objective was used to increase the resolution, the depth of field would correspondingly decrease. The advantage of the depth of field available with the SEM is readily visible.

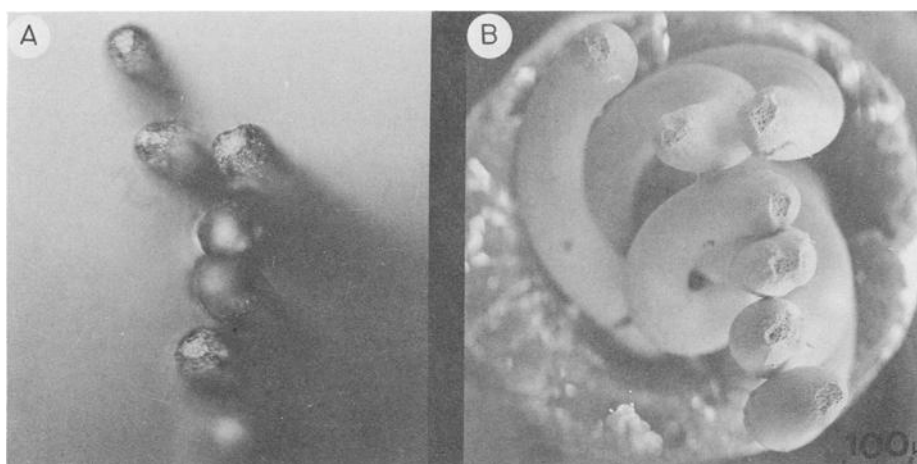


FIG. 13—Comparison of a light micrograph (a) and an electron micrograph (b) of the same multistranded wire broken in tension.

Conclusions

Given specimens whose broken surfaces are to be compared, a good light microscope should be the first tool to be applied in the examination. If a matching of fractures can be performed, then no further steps need to be taken. If, however, because of resolution or depth of field limitations, a required comparison is not possible, the scanning electron microscope with the auxiliary X-ray analyzer can be employed in the investigation.

As a general rule, the SEM can be used to good advantage in examinations where the fracture surfaces have a dimension smaller than $50\ \mu\text{m}$ (0.002 in.). The SEM can also be useful where larger items had been severed by a cutting tool. In metals cut by any type of tool, including saws, the final separation of the metal occurs by fracture. These fracture areas can be extremely small, but nevertheless may be adequate for SEM fracture comparison.

If an examination with the SEM is indicated, a fast (one-minute) X-ray analysis of the specimen is the next logical step in the examination. A qualitative discrepancy between the broken parts would normally preclude any matching possibility. Care should be

taken that both surfaces are free of contaminants (or have equal exposure to the same contaminant) before undertaking this step.

If a rough qualitative match exists in composition, the examiner may now choose between a more quantitative analysis or a comparison of the break surfaces by scanning electron microscopy. This decision is one which depends on the specimen, the examiner, and the type of equipment available.

In mounting the specimen in the SEM, use should be made of the information obtained during the preliminary optical examinations, especially regarding orientation and alignment. A preliminary comparison of both halves of the break surface should now indicate the area of possible match, the optimum magnification, the accelerating voltage, and the angle of viewing to be used for comparison.

Broken metal wires can be matched by an SEM examination of the fracture surfaces. The probability of matching two halves of a broken wire will depend on the way the break was formed and on the amount of contamination and distortion the surfaces have acquired since the break occurred. Once contamination enters a deep pore, very little can be done without the use of some "harsh" treatment [6].

References

- [1] Phillips, A., Kerlins, Y., and Whiteson, B. V., "Electron Fractography Handbook," AFML-TR-64-416, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Dayton, Jan. 1965.
- [2] McCall, J. L., "Fracture Analysis by Scanning Electron Microscopy," MCIC-72-12, Battelle Columbus Laboratories, Columbus, Dec. 1972.
- [3] Meyn, D. A. and Beachem, C. D., "Precision Matching of Mating Fracture Surfaces in the Electron Microscope" in *Proceedings of the 7th Annual SEM Symposium*, O. Johari and I. Corvin, Eds., IIT Research Institute, Chicago, 1974, pp. 903-910.
- [4] Rogers, H. C., "The Mechanism of Crack Propagation in Ductile Metals," *Acta Metallurgica*, Vol. 7, No. 11, 1959, pp. 750-752.
- [5] Brown, J. L. and Johnson, J. W., "Electron Microscopy and X-Ray Microanalysis in Forensic Science," *Journal of the Association of Official Analytical Chemists*, Vol. 56, No. 4, 1973, pp. 930-943.
- [6] Dahlberg, E. P., "Techniques for Cleaning Service Failures in Preparation for SEM and Microprobe Analysis" in *Proceedings of the 7th Annual SEM Symposium*, O. Johari and I. Corvin, Eds., IIT Research Institute, Chicago, 1974, pp. 911-918.

FBI Laboratory
Washington, D.C. 20535