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MEMORANDUM REPORT ARBRL-MR-02980

TECHNIQUES FOR DETECTING CARBON FIBER CONTAMINATION ON SURFACES

William F. McBratney

January 1980

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US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
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Several methods are described for detecting and locating carbon fibers on relatively flat surfaces. Ultraviolet and visible light techniques are used to visually examine wetted surfaces. Variations in the surface curvature or thickness of the wetting film allow improved detection. Specular reflection and contrast may also be used for the detection and location of carbon fibers.

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I. INTRODUCTION

A major goal of the protection effort in the JTCG HAVE NAME program is the identification of cost-effective techniques to decontaminate potentially harmful carbon fibers (CF) from military systems and equipment. A necessary part of this effort requires the development of methods for detecting and identifying CF contamination in equipment.

This study specifically addresses the evaluation of alternate techniques for detecting carbon fibers on surfaces. The results will be used to aid in conducting decontamination tests in the laboratory and possibly for detecting carbon fibers on surfaces under "field" conditions.

The carbon fibers under consideration are the carbon/graphite fibers that are being used in structural composites. The fiber diameter is on the order of eight micrometers.

For the purpose of this task, techniques were desired that required minimal investment (time and financial) and that were readily available. A number of techniques were considered; on the basis of simplicity and utility, techniques that utilize the properties of films of liquids, blacklight inspection techniques, and contrast enhancement were evaluated.

II. DISCUSSION

The "standard" technique for counting fibers on surfaces has been the sticky tape technique. A sticky, transparent plastic applied to the surface traps the fibers. The "tape" is removed and applied to a slide frame. The slide is then projected on a white surface and the magnified fibers are counted.

Depending on the characteristics of the surface, low power magnification has been utilized for fiber detection. For surfaces of moderate contrast to the fiber, magnification is beneficial. For dark, shiny surfaces, low power magnification is not of much help, due to the poor contrast.

In considering the possible additional methods of detecting and counting the fibers on surfaces, simplicity in technique and equipment was desired. This emphasis directed the effort along the lines of enhancing the visibility of the fibers. The following section discusses the simplest resulting techniques. These techniques are based on (1) contrast enhancement, (2) black light inspection techniques, and (3) the properties of applied surface films.

Other techniques were considered but not developed. Holographic, IR, and dye/stain techniques are briefly considered in Section IV.

III. TECHNIQUES OF VALUE

A. Specular Reflection From Carbon Fibers

The quickest method of detecting carbon fibers on a surface is to look for the bright specular reflection from the fiber. This reflection corresponds to the mirror-like reflection that may be observed from a highly polished cylinder.

A flat plate contaminated with carbon fibers is laid on a table, and is illuminated with a bright light. A viewing position is selected that avoids the reflection of the plate surface. The plate is then observed while it is slowly rotated on the table surface. Bright, line-shaped reflections will be observed from the fibers for well defined positions of the plate.

The fiber may be treated as a long, thin mirror. The specular reflection corresponds to looking at the light source in this mirror. The angle through which the specular reflection may be observed depends upon the fiber length, the width of the light source, and the viewing distance. With a small light source and a long fiber, specular reflection may be obtained from only a part of the fiber at a given orientation.

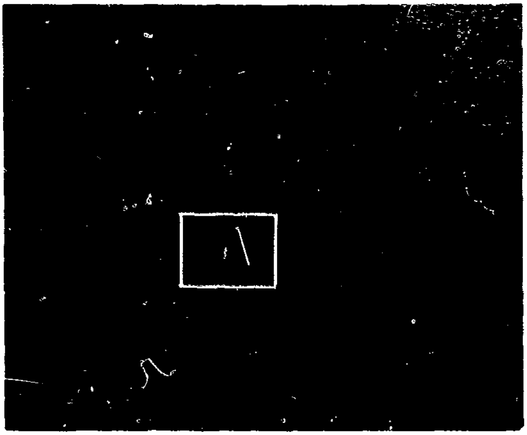
Figure 1 shows a 35mm slide cover glass upon which two fibers were placed. The fibers are crossed and oriented at approximately 30° to each other. The light is incident from the direction roughly perpendicular to the axis of the visible fiber and above the plane of the slide. Clearly, the only fiber that is observable is the fiber that meets the orientation requirements for observing specular reflection.

The major defect with this technique is that fine scratches in metal surfaces and some plastics may also give brilliant reflections from the sides of a scratch. These are difficult to distinguish from the carbon fiber reflections.

B. Ultraviolet Inspection of a Wetted Surface

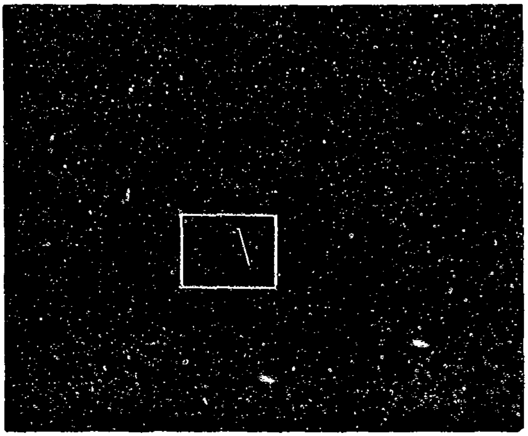
Black light inspection techniques are in normal use in a number of areas.

Magnaflux testing is a technique for detecting cracks and defects in metal structures (especially tubes and rods). A liquid containing fluorescent magnetic particles is sprayed onto the metal part while a magnetic field is generated in the part. Cracks and defects distort the magnetic field and trap the magnetic particles. Inspection with a black light locates the luminous area.



Two 7.5mm HMS fibers, crossed, 30° difference in orientation, illumination incident perpendicular to the observed fiber.

Figure 1. Specular Reflection from Carbon Fibers.



Two 7.5mm HMS fibers, crossed, 30° difference in orientation, illumination incident perpendicular to the observed fiber.

Figure 1. Specular Reflection from Carbon Fibers.

Oxygen handling equipment is routinely inspected using black light to detect the presence of traces of oily materials. Oils and greases in contact with pure oxygen are a fire and explosion hazard.

Standard type black light inspection lamps, e.g. filtered flood lamp type and the fluorescent tube type fixtures of high output, were obtained in order to test various possible black light detection techniques for carbon fibers.

Preliminary tests indicated that many readily available oils fluoresced brightly enough to be readily visible in a darkened room and that their film thickness could be reduced to values less than the diameter of test carbon fibers, approximately eight micrometers.

Light "oils" [WD 40, penetrating oil, light instrument oil] were sprayed or spread on carbon fiber contaminated glass slides and allowed to drain to a thin layer. The wetted fiber on the wetted surface caused a thicker layer of oil to be held near the fiber. Inspection with black light shows the thicker layer of oil as a brighter region. Figure 2 shows three fibers on a 35mm slide cover glass wetted with penetrating oil. The bright zones along the fiber sides are readily visible above the background fluorescence. Figure 3 is an illustration of the film thickening near the fiber.

During the tests with thin layers of penetrating oil and light instrument oil, the fibers were observed to cause bright zones along the fiber in layers of oil thicker than the fiber diameter. Inspection of the oil surface revealed no surface curvature such as would be associated with a meniscus.

The explanation for this increased luminosity around the fiber is that some ultraviolet light is reflected from the fiber (Figure 4). The literature (Reference 1) indicates that on the order of 30% of the light incident perpendicularly to the basal plane of a graphite crystal will be reflected. Normally, as the angle of incidence deviates from perpendicular to a surface the reflectivity increases. For highly graphitized carbon fibers, the sides of the fiber correspond to the basal plane of a graphite crystal. This means that more than 30% of the incident ultraviolet light should be reflected into the region around the fiber.

The visible light emitted under fluorescent emission is generally directly proportional to the intensity of the ultraviolet stimulating radiation. For the thin layers of oil used in these tests, the increase in stimulating radiation due to reflection was enough to make the fibers visible against the neighboring regions.

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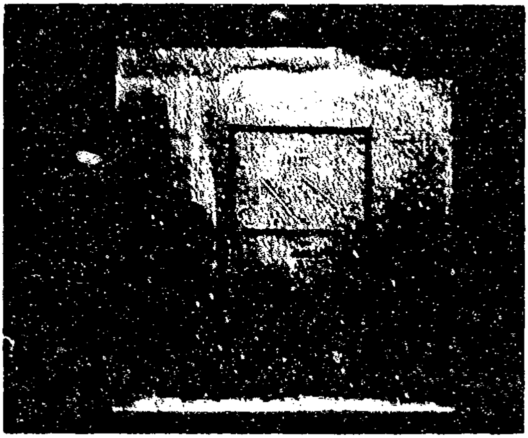
[1] *W. N. Reynolds, "Physical Properties of Graphite", Elsevier, Amsterdam, 1968.*



Three 7.5mm HMS fibers on a glass plate wetted with penetrating oil, magnification 1.4X.

Figure 2. Ultraviolet Inspection of a Wetted Surface.

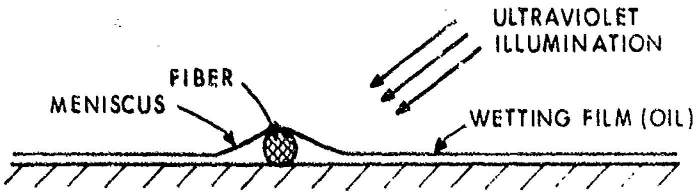


Figure 3. Illustration of Film Thickening Near a Fiber on a Wetted Surface.

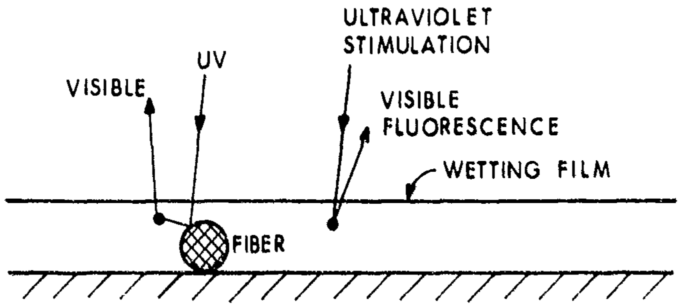


Figure 4. Fluorescence Intensity Enhancement by Reflection from the Fiber.

Several problems are associated with the fluorescence (black light) techniques. Some of the materials encountered under field conditions are likely to be rough enough or naturally fluorescent enough to cause interference with the visual detection of carbon fibers.

C. Visible Light Illumination, Wetted Surface

A "flat", polished surface reveals small surface defects or contamination when the surface is held towards a light source. Similarly, a painted surface will show "bumps" or fibers that are appreciably larger than the film thickness.

A wetted surface will appear mirror smooth if the wetting material is thicker than the "tallest" irregularities. By varying the thickness of the wetting material, various "terrain" features may be brought to the surface and cause distortion of the mirror-like surface.

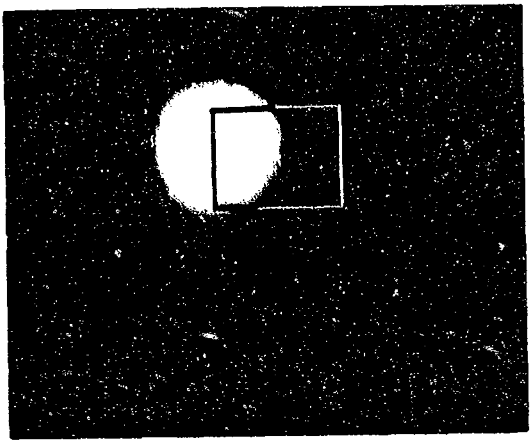
Figure 5 shows three HMS fibers on a glass slide which has been coated with a thin layer of penetrating oil. The thickness of the oil layer is such that the fibers cause a broad disturbance in the layer thickness in their vicinity, and cause a curvature of the surface. Light incident upon the meniscus near the fiber will be reflected in a different direction than the light incident upon the flat surface. Thus if the contaminated surface is examined in a beam of light, bright reflections will be noted in directions away from the main beam reflection angle and darker zones will be noted in the main beam reflection direction.

For practical applications, a diffuse light source, such as a low wattage frosted bulb or a tissue paper covered flashlight, was found to be best. Looking towards the reflection of the light source on the surface, bright lines are observed slightly away from the major bright patch and dark lines are visible within the edge of the bright patch. These lines correspond to the altered reflection angles for the light incident on the slightly sloping liquid surface near the fibers.

Wetting agents are available (detergents) that will allow water or alcohol based liquids to be used in this application. This allows the liquid to evaporate, leaving very little surface contamination after the inspection. Some of the organic solvents would also serve as a volatile, low residue base for this inspection technique.

D. Contrast Enhancement

Single carbon fibers are readily visible against highly contrasting backgrounds. The specular reflection from a carbon fiber provides a bright reflection. However, the small diameter of the fiber causes this reflection to be a low level visual signal that can be difficult to de-



(Three 7.5mm HMS fibers penetrating oil wetted surface)

Figure 5. Visible Light Illumination of a Wetted Surface Showing Surface Defects.

tect against a "bright" background. Similarly, the small size of the fiber causes detection difficulties when color contrast is depended upon unless the contrast is marked.

Against a matte black background, the specular reflection from a carbon fiber produces a high visibility object.

A diffuse white surface of high reflectivity provides a contrast with carbon fibers that is detectable at normal reading distances. The contrast in this situation does not require the specific orientation that is required by the specular reflection technique.

For laboratory type of tests requiring the detection of residual carbon fiber contamination, the surface to be tested should be selected to provide high contrast if possible. Various techniques are available to blacken metal surfaces, such as sulfide treatment, etching, and anodization.

IV. ADDITIONAL TECHNIQUES CONSIDERED

In addition to the techniques discussed in the previous section, several techniques were considered but not pursued. These are mentioned here as approaches only.

A. IR Emission

The blackness of the fibers implies a high emissivity. This could be utilized in an infrared detection system (photographic). By heating the inspection surface, the higher emissivity of the carbon fiber would be apparent as a brighter area relative to lower emissivity surfaces.

The absorption of microwave radiation by carbon fibers acting as dipole antennas, could be used to heat the fibers for IR detection or visible range detection. The strong dependence of the absorption upon fiber length would complicate this procedure for any but the most restricted tests (Reference 2).

[2] *Joseph D. Adams and J. Lee Edwards, "Investigation of Electrical Properties of Materials (U)", RADC-TR-71-150, August, 1971. (Secret)*

B. Holography

A complicated, but interesting, approach to detection is through holographic technology. A given object may be used to generate a holographic pattern. The image from the area of interest may then be optically processed in comparison to the reference pattern as a target detection technique (Reference 3). This technique was considered to be beyond the complexity level desired in this effort.

C. Dye/Stain

As a result of discussions with W. Beims of this laboratory, the use of dyes in a detection technique was given some initial consideration. Sticking of the dye to the fiber itself offers little apparent advantage, however a potential exists for detecting color intensity variations in regions adjacent to a fiber. This color intensity variation could be achieved in liquid films containing the dye for films that are thinner than the fiber diameter and for liquids that wet both the fiber and the substrate. As the film thickness decreases below the fiber diameter, a thicker liquid layer will be present along the fiber due to the surface tension of the liquid. The thicker zone will have more intense coloration. This approach requires a dye that contrasts with the substrate color. Also, the concentration of dye in the liquid must be adjusted to give a strong difference in intensity in films a few (one to ten) micrometers thick.

An alternate approach would be to use a volatile dye carrier. In this approach, a layer of dye solution is allowed to evaporate. The zone around the wetted fiber will again contain a thicker layer of liquid. As the evaporation continues, the dye becomes more concentrated in the liquid. The final thick zone along the fibers will act to deposit the concentrated dye on the substrate. Examination of the dry surface should indicate the location of the fibers by the local strips of concentrated dye.

False signals would be produced by either of these dye techniques at scratches. The uneven drying of the volatile liquid also produces irregular concentrations of dye.

In light of the successful black light technique discussed earlier, this dye approach was not pursued to a recipe stage.

[3] *George W. Stroke, An Introduction to Coherent Optics and Holography, Academic Press, New York, 1966.*

D. Spark Mapping

A method for rapidly locating conducting fibers on insulating surfaces was tested briefly. An alternating high voltage probe, as used in detecting leaks in glass vacuum systems, was adjusted to give a small corona discharge. When the probe was brought near (several centimeters) a conductive fiber, sparking was observed from the ends of the fiber. This is due to the distortion of the electrical field of the probe by the conducting fiber causing the electrical break down of the air.

As the probe is brought nearer the fiber, sparks are observed to jump from the tip of the probe to the fiber.

This technique, as tested, has several drawbacks. The corona around the tip of the probe causes the air to become ionized. In the tests, some of the fibers were observed to be "blown" from the surface as the probe approached.

With the system tested, sparks would be drawn to random uncontaminated areas of the insulating surface thus giving false signals. The identification of the spark terminus as a fiber had to be confirmed by other visual means.

This system could probably be improved by redesigning the electrode system to reduce the corona effect and the drawing of sparks to the surface. By controlling the electrode shape and the distance to the inspection surface a characteristic sparking at the ends of fibers should be obtainable.

It should be emphasized that this technique is limited to nonmetallic surfaces exclusively and that catastrophic damage of sensitive electronic components could result from using this method.

V. SUMMARY AND RECOMMENDATIONS

Visual detection of fine fibers is strongly dependent upon the observer being familiar with the visual characteristics of the fiber being sought. Anyone wishing to detect carbon fibers should acquire test samples of the material and become familiar with their characteristics under various test conditions. Visual characteristics such as reflectivity, color, and straightness due to high mechanical modulus provide clues to detection that are difficult to verbalize adequately but readily observable in samples.

For laboratory tests, the contrast enhancement approach of selecting the surfaces for contrast with carbon fibers provides the simplest approach. The method of wetting the surface with a thin layer of wetting material and inspecting for surface irregularities in the otherwise flat surface film provides another simple, rapid procedure. The black light inspection technique is a usable technique but generally requires more time and caution.

Detection of carbon fibers on surfaces under "field" conditions presents many conditions that interfere with visual detection techniques. It is necessary that the inspector be aware of the conditions that interfere with or enhance a particular method. Inspection of the surface in a darkened enclosure allows the use of a single source of illumination and improves the relative signal from a fiber. Where a light oil (e.g. WD 40) may be sprayed, distortion of the surface film may be observed under diffuse illumination. Specular reflection techniques are useful when the surface is exposed so that illumination and observation directions may be varied over large angles, but a brightly colored, glossy surface yields reflections that are strong enough to interfere with this technique. Microscope or fiber optic illuminators provide compact, intense light sources that are useful in these techniques, however normal flashlights of various sizes are useable. Diffuser screens or tissue paper may be used on these light sources. The black-light inspection technique may be used if the work area can be darkened. A high intensity projector spot type ultraviolet lamp (e.g. VWR #36550-047) was found most useful in these tests.

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