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MICROENCAPSULATED FLUORESCENT DYE PENETRANT. (U)

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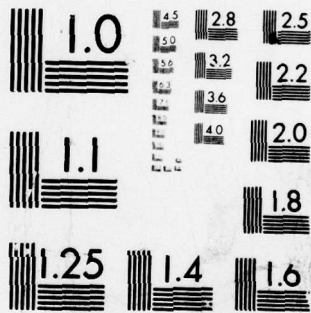
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MICROENCAPSULATED FLUORESCENT DYE PENETRANT

Non-Metals Application Branch  
Systems Support Division

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July 1979

TECHNICAL REPORT AFML-TR-79-4027

Final Report for Period October 1977 - January 1979

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Many of the materials tested were not developed or intended by the manufacture for the conditions to which they have been subjected. Any failure or poor performance of a material is, therefore, not necessarily indicative of the utility of the material under less stringent conditions or for other applications.

This report has been reviewed by the Information Office (IO) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nations.

This technical report has been reviewed and is approved for publication.

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FOREWORD

The experimental work reported in this text was conducted by the Air Force Materials Laboratory, October 1977 through January 1979. The work was performed under Job Order No. 24210302, Specialty Materials Applications for Systems, and was administered under the direction of the Systems Support Division, Air Force Materials Laboratory, Air Force Systems Command, Wright-Patterson Air Force Base, with Mr. S. Allinikov (AFML/MXE) as Project Engineer.

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SECTION I  
INTRODUCTION

Fluorescent penetrant inspection is the most widely used nondestructive inspection (NDI) method within the Air Force Logistics Command's Air Logistics Centers. Also it is widely used within industry. While certain improvements in penetrant formulations have been made over the years, the penetrant inspection process has remained unchanged (Reference 1). Parts are cleaned, immersed in penetrant for some time period, e.g., 20 minutes, the part is then cleaned to remove all penetrant except that contained in defects, developer is applied to widen the penetrant indication, and the part is inspected under ultraviolet light.

The removal of the excess penetrant in some cases is accomplished by water washing. Some of the more sensitive penetrants must first be emulsified to render those water washable, and then water washed. After this step is completed the part is dried, and developer is applied, and inspection is then done under ultraviolet light. The process of applying penetrant, washing, drying, and developing can take as long as an hour.

Over the years users of penetrant have expressed concerns with this process. Excessive washing can result in removal of penetrant from defects. The application of emulsifier must be carefully controlled. When parts containing penetrant are moved into the emulsifier tank the emulsifier eventually becomes sufficiently contaminated with penetrant, and the emulsifier must be disposed of and replaced with fresh emulsifier.

Inspection must be accomplished within a specific time period after developer is applied. If this period is exceeded the fluorescent indication is lost.

Penetrant materials can consist of penetrant, emulsifier, and developer. These materials or solutions are not interchangeable between the various penetrant manufacturer's products. That is, one company's emulsifier cannot be used on another company's penetrant. The penetrant materials

must be used as a "family." This can and does become a supply and logistics problem.

These aforementioned problems led to this idea of microencapsulated penetrants.

A fluorescent solution can be microencapsulated to create very tiny plastic spheres containing the fluorescent solution. In effect we now have a "dry" fluorescent material. When sprayed the mechanical force would force these small fluorescent particles into defects. The rest of the particles would bounce off of the surfaces of the parts to be inspected or would be so loosely held as to be easily removed.

Thus we had envisioned a "dry" process which would require just a short time to accomplish, and not involve any time critical steps, nor a family of chemicals.

The ensuing parts of this report will describe the work done using microencapsules containing various dyes prepared by the 3M Company, and 2-5 micron diameter spheres made of a flexible plastic and containing Zyglo ZL-30, a Group VI penetrant meeting Spec. MIL-I-25135. These tiny spheres are easily deformable. The use of Zyglo ZL-30 was a matter of convenience since it was already in wide use and is known and accepted as a sensitive, bright penetrant. In reality one may need only to dissolve a fluorescent dye in any encapsulatable liquid. It does not have to be a product formulated as a liquid penetrant to meet MIL-I-25135.

The microencapsulation of the Zyglo ZL-30 was performed by Encapsulated Systems Inc., Yellow Springs, Ohio under an Air Force contract. In the process of microencapsulation a small portion of the microcapsules can be cracked and leak dye. However in procuring microencapsulation one can specify that the product be washed. This removes any leaking dye so that the delivered microcapsules would not include such "leakers".

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At the time we procured the microencapsulated products we were unaware of this and the product we worked with had not been washed and thus could have contained a small amount of such leakers. A small quantity of free dye can cause clogging of the deposition apparatus, and also make removal of excess background fluorescence more difficult. Therefore, the capsules should be washed as a matter of course to prevent this problem.

SECTION II  
EXPERIMENTAL

1. INITIAL FEASIBILITY EXPERIMENTS

Several experiments were conducted to determine feasibility of the use of the fluorescent microcapsules to detect flaws on metal surfaces. The initial experiment consisted of spraying a quantity of the dry microcapsules on an engine turbine blade containing a large crack. The crack dimensions were not measured. A laboratory type paint spray gun was used to apply the material to the blade surface. Figure 1 is a photomicrograph of the capsules. These capsules ranged from 8 to 16 microns in diameter and contained a Rhodamine B dye solution. The excess capsules were removed from the turbine blade with a dry cotton swab. The capsules were easily removed except at the crack location, where these had evidently lodged in the crack. Figure 2 shows the capsules lodged within the crack on the turbine blade. The fluorescent indication was highly visible under ultraviolet irradiation.

2. CRACK STANDARDS

These test results were sufficiently encouraging to warrant further tests. These were accomplished on crack standards furnished by the Materials Integrity Branch (AFML/MXA) within the Materials Laboratory. Two types of standards were used in the tests. The first type was a set of three chrome-plated brass crack standards. These panels are prepared from polished brass stock and are plated with nickel and then with chrome. Dimensions of the panels are 10 cm X 6.7 cm. The plating and cracking procedure varies with the type of crack standard desired, (course, medium, and fine cracks). Electrographic prints are made of the cracked panels as a reference in determining sensitivity of the penetrant applied to the crack standard. A complete description of the preparation of the standards is given in technical report, AFML-TR-65-64, "Fluorescent Penetrant Inspection Materials Evaluation Method Development and Comparative Values, by J. C. Harris of the Monsanto Research Corporation. Each of the three panels represented a specific maximum crack width.

Crack widths were 12.7, 3.5, and 0.5 microns respectively for the coarse, medium, and fine crack standard panels. Figure 3 is representative of the MRC standard.

The other standards used were made of steel bars with dimensions of 15.6 cm X 2.5 cm. The cracks were prepared by creating a "dimple" in the surface of the metal with a defocussed laser beam. The steel bar was then placed under three point loading in a fatigue test machine to produce a fatigue crack. The bar surface is milled to remove any radial cracks which might have occurred from "dimpling" prior to insertion of the piece in the fatigue apparatus. The crack width of each of the two steel standards furnished were 0.5 and 2.5 microns respectively. These dimensions were determined by optical measurements, made with a Unitron ME-1945 metallurgical microscope. Figure 4 shows the two steel standards prior to penetrant application.

### 3. CLEANING OF CRACK STANDARDS

The cleaning procedure schedule applied to the MRC standards prior to deposition of the capsules and inspection of the standard is indicated in Table 1.

TABLE 1  
REVISED CLEANING PROCEDURE

<u>Step</u>	<u>Time</u>
1. Hot water wash (running water)	1 min.
2. Wipe surface with clean soft towel	
3. Hot water wash (running water)	14 min.
4. Boiling distilled water	30 min.
5. Forced hot air dry	5 min.
6. Evacuate in vacuum dessicator	15 min.

The steel panels were cleaned in an ultrasonic bath containing a detergent. The panels were then dried with forced hot air after a distilled water rinse.

#### 4. MICROCAPSULES

Three different dye solutions and one commercially available fluorescent dye penetrant solution were microencapsulated. The dyes encapsulated were Rhodamine B, Fluorol Yellow, and a substituted naphthalamide compound. The encapsulated penetrant solution was Zyglo ZL-30. Diameters of the capsules containing the dye solutions ranged from 1 to 10 microns for the naphthalamide dye, 3 to 11 microns for the Fluorol Yellow, and 8 to 16 microns for the Rhodamine B. These materials were microencapsulated by the 3M Company during our initial efforts to determine feasibility of this project. The 3M capsule wall was a urea-formaldehyde polymer. The Zyglo ZL-30 dye penetrant was microencapsulated by the Capsulated Systems, Inc., Yellow Springs, Ohio. The capsule diameters range from 2 microns to 5 microns. The capsule wall is a phenolic material and is approximately 0.5 micron thick. Both suppliers furnished the material in dry powder form at our request. The ZL-30 was selected for microencapsulation because it was a qualified Group VI, MIL-I-25135 penetrant and had high brightness. The other dyes in solution previously mentioned are bright, but additional development work would be needed to provide a dye or combination of dyes with the brightness of the Zyglo ZL-30, already available.

#### 5. DEPOSITION OF MICROENCAPSULATED PENETRANT TECHNIQUES

Deposition of the microcapsules on to the test surfaces was accomplished by several different techniques as follows:

- a. Physically rubbing the material over the surfaces with a cotton swab.
- b. Spraying the capsules through a laboratory size paint spray gun. Air pressures from 25 psi to 80 psi were used to deposit the capsules onto the test surfaces. The spray gun (Model EGA) was made by the DeVilbiss Company and is equipped with a F size needle and fluid tip, and #390 air cap. A 4 oz. glass cup contained the capsules to be sprayed.

c. An air eraser spray apparatus was also used for the experimental deposition of the capsules. This device made by the Paasche Airbrush Company is normally used to spray a dry abrasive powder by artists for removal of ink lines and by dental technicians for cleaning dental prosthetics. Nitrogen pressures were used in the ranges of 10 psi to 40 psi for application of the capsules. Pressures above 25 psi showed the most useful effects in the case of the air eraser.

d. The microencapsulated materials were also applied by a PRE-VAL aerosol spray unit of the type available in hardware stores. The aerosol assembly consists of a 6 oz. capacity glass container and a replaceable "power pack" which contains a propellant, siphon tube, and spray nozzle.

e. The microencapsulated materials were applied dry in all experiments with the exception of one trail with the aerosol spray. In this case the microcapsules were dispersed in methyl ethyl ketone, and sprayed on a Monsanto type fine crack standard.

#### 6. REMOVAL OF MICROCAPSULES

Several methods of removal of the excess microencapsulated materials not lodged in the defects and cracks to reduce background fluorescence were explored. These included dry and wet removal methods. Removal of the capsules by the dry methods was attempted with cotton swabs, medium bristle brushes, concurrent brushing and vacuum, air pressure, and by vacuum alone. In addition a rinse with a detergent-water solution was evaluated. The latter included the rinse alone, and a rinse with gentle scrubbing with a medium hard bristle brush (acid cleaning brush).

#### 7. INSPECTION PROCEDURE

The test pieces were visually examined for indications of porosity or cracks under an ultraviolet lamp. Specimens that have been subjected to a dry removal of capsules were inspected with no further removal processing. The crack standards which had been wet cleaned were dried with a lint free paper towel prior to inspection. The pieces were visually

inspected for crack indications with the aid of an ultraviolet lamp. The parts were inspected both before and after the application of a commercial developer. This was done to determine the possible effect and relative value of a developer in enhancement of the crack indication.

SECTION III  
DISCUSSION OF RESULTS

1. INITIAL EXPERIMENT

The first feasibility experiment (Figures 1 and 2) indicated that the microcapsules could be deposited on a surface by conventional equipment and techniques. It also showed that capsules would lodge in a crack, and the excess material could be easily removed to leave a crack indication with little or no background fluorescence. It was also noted that the capsules did not break upon impact with the test surfaces. Although the capsule walls are only 0.5 to 1 micron thick, they can usually withstand high mechanical loads without breaking or releasing liquid contents. The indication of the crack was bright and definitive, however this was not surprising because the defect was quite large. Removal of the microcapsules once lodged in the crack was difficult. Ultrasonic cleaning was usually required to remove those capsules from the crack. Of course this was desirable, since it shows the capsules and the flaw indication are not easily obliterated.

2. APPLICATION METHODS

Deposition problems were encountered with the urea-formaldehyde microencapsulated dye solutions when applied as dry powders from the different pieces of spray apparatus. The principle difficulty experienced was intermittent clogging of the equipment, which occurred regardless of the application spray pressure. This was attributed to the presence of some free dye solution on the surfaces of the capsules. The free dye solution may have migrated through a porous capsule wall, or perhaps was due to the presence of capsules broken during polymerization of the wall material. A very small percentage of the liquid can cause clogging of the spray apparatus. It is very important to specify that the microcapsules be dry, washed, nonporous, and free flowing in order to prevent this problem with the material in dry powder form. The capsules could be physically rubbed into cracks, however and provide good indications. This deposition method may be useful for parts with flat uncomplicated surfaces. It does not appear to be feasible for most inspection tasks.

The microencapsulated Zyglo ZL-30 material with capsule diameters under 5 microns supplied as a dry free flowing powder could be applied much more easily by any of the deposition methods. Penetration of the cracks and indications were achieved by all methods on the Monsanto (MRC) type Standards. The aerosol and paint spray gun provided a more uniform distribution of the capsules over the surface compared to the eraser. This can be seen in Figures 5 and 6. Figure 5 shows capsules applied by spray on an MRC standard prior to removal of excess capsules. Figure 6 is the same standard with capsules applied by the eraser gun. The latter apparatus provided a relatively narrow concentrated spray pattern. The aerosol spray device has no means of adjusting nozzle pressures, therefore is not as versatile as the paint spray or air eraser units. It undoubtedly can be valuable for inspection of some parts installed on aircraft. A paint spray gun or other spray apparatus which delivers a relatively wide spray pattern with a pressure regulator is the most desirable deposition method based on tests made to date.

Variations in pressures appeared to have some effect relative to penetration of the crack standards based on observed variation in brightness of the indications. This was somewhat difficult to ascertain by visual observation since there were no radical differences in brightness with varying application pressures. Changes in the brightness of indications were made noticeable between pressure changes up to 40 psi at the spray nozzle. Application pressures above 40 psi did not enhance brightness. Consumption rate of the capsules applied as a dry powder was not measured, but appeared to be rapid, especially when sprayed with the paint spray gun. Most of the material was lost as overspray, which would be readily recoverable with proper equipment.

The single experiment where the capsules were dispersed in methyl ethyl ketone and sprayed on the surface of an MRC fine crack standard indicated the material could be deposited this way if desired. The crack standard was uniformly covered with capsules, and the cracks could be readily observed upon subsequent inspection. The solvent flashed off rapidly so that the workpiece surface was dry almost immediately after the spray application.

### 3. REMOVAL OF EXCESS CAPSULES

In general, removal of excess capsules was dependent upon surface roughness of the inspection item. Relatively smooth surfaces did not present a problem in dry or wet removal of the excess capsules. The fluorescent background was very low and cracks in the standards could be discerned readily. Rougher surfaces, especially in pitted areas did present a capsule removal problem with the dry removal techniques. The most efficient dry capsule removal method appeared to be a combination of mechanical brushing concurrent with a vacuum cleaning action.

The best results in removing the background fluorescent capsules were obtained by a rinse with the detergent-water solution prior to inspection. This method worked very well on smooth and rough surfaces. The wet capsule removal process was rapid and not sensitive as to the time of removal after the capsules had been deposited on to the test surfaces. It is probable that a static charge was developed on the capsule surfaces, thus making it more difficult to remove by dry methods. Some tests in which appropriate charging of the penetrant capsules or of the part being sprayed may cast more light on this aspect.

### 4. MICROCAPSULES

Test results indicated that size of the capsule is not necessarily indicative of the crack widths that can be entered. It is true that with capsules which are smaller than the cracks they are impinged upon, entry into the cracks will occur. It was also found that capsules (8-16 microns) larger than the crack widths provided indications on the coarse, medium, and fine MRC crack standards. The mechanism by which this occurs is not clear at present, since no attempt such as microscopy was made as yet to investigate this observation. The capsule walls are flexible, and could wedge into a crack when applied with a pressurized spray apparatus. The capsules may break on rough crack edges, and allow the penetrant to flow into the crack. It is also possible that only small capsules enter the cracks, because of capsule size distribution in any given batch of capsules. The majority of capsules will be within the size range specified, but smaller capsules will usually be present

in the batch. It is possible to encapsulate and then isolate capsules of a narrower size range, but this is a more costly process and it does not appear to be necessary.

The smaller diameter capsules are most easily isolated when dispersed in a liquid. Dry capsules, however, were specified for this application, which poses somewhat of a problem in isolation of the individual capsules. They tend to agglomerate, probably because of static attraction. Figure 7 provides a 160 X magnification view of the capsules in agglomerated form. The clusters of agglomerated capsules are shown clearly in Figure 8. The capsules are viewed at 4200 X magnification in this photograph. It was found that the agglomerated capsules separated upon impact with the workpiece and therefore indicated only a weak attraction exists between the spheres. This attraction may be alleviated by different drying techniques or by the addition of an anti-static coating to the capsule walls. The micrographs for Figures 7 and 8 were generously provided by the Hughes Aircraft Company, Culver City, California.

## 5. INSPECTION RESULTS

The major effort on this project was accomplished with the 2 to 5 micron encapsulated Zyglo material for the reasons previously discussed. The crack indications could be readily discerned in all three MRC standards under an ultraviolet lamp. The fine crack standard is shown in Figure 9. The corresponding electrographic print (Figure 10) for that standard is shown to indicate the sensitivity of the microcapsules. The fluorescent indications very clearly are identical to the lines in the electrographic print.

These capsules also penetrate or indicate the 2.5 micron width fatigue crack as illustrated in Figure 11, and also the 0.5 micron width fatigue crack shown in Figure 12. These samples were all washed with the detergent-water solution and then dried prior to inspection. They were treated with Met-L-Check D-70 spray developer before inspection, since brightness of the indication was enhanced in these cases by use of the developer. The role of the developer in improving visibility of the

indication is still unclear. It is believed that on shiny surfaces the developer powder may be reducing visible light reflection of the background surface, thus improving contrast. In some instances, the capsules may break upon impact on sharp edges of a crack. This would allow the developer to "wick" the free fluid in the manner it is alleged to work with conventional liquid penetrant formulations. The capsule wall material did not appear to be affected by the solvent in the developer, which could explain the erratic results experienced. The developing agent probably does not improve contrast if the capsules are intact and reflection from the background surface is not a problem. It is not difficult to use a microencapsulation material that is attacked by the carriers in commercial developers if that becomes a requirement to improve visibility of the crack indications. Inspection of the standards again one week after deposition of the capsules revealed the indications were still visible. No additional processing was required from that done the previous week.

SECTION IV  
DISCUSSION AND SUMMARY

The microcapsulated fluorescent materials show feasibility for application in detection of cracks on metal surfaces, although the process need not be restricted to metal. The material can be applied dry as a free flowing powder or in a dispersed form in water or other solvents. It has been demonstrated the microencapsulated Zyglo ZL-30 material will show 0.5 micron width cracks on metal even when applied as a powder. It is imperative to specify nonporous, washed, free flowing capsules for dry deposition of the material. This will ensure the lowest background fluorescence and allow the material to be sprayed or otherwise dispensed without clogging the deposition equipment.

The microcapsules seem to be totally insensitive to processing variations in application and removal prior to inspection than are the liquid penetrants. The processing time prior to inspection is much shorter than with the conventional post emulsifier systems. For example in the experiment described herein the time period from spraying the encapsulated penetrant to inspection was a minute or two. The microcapsules contained in the overspray during dry deposition can be readily recovered and reused. If the capsules were to be applied dispersed in a fluid, contamination of the supply reservoir should be decreased. The capsules could also be reclaimed after removal if it proved necessary to use a detergent-water wash. The microcapsules can contain fluorescent compounds that might not otherwise be useable today in liquid penetrants because of toxicity problems. It should definitely reduce handling problems from the health standpoint. The microcapsules are very readily removed from the hands by a simple soap and water wash.

The difficulty in dry removal of excess capsules from the test pieces may be due to static attraction of the capsules to the substrate surfaces. Various schemes for deposition and removal of the capsules to minimize this problem should be investigated. Examples would be to appropriately electrically charge the capsules and/or parts to be inspected or to apply

an antistatic coating or agent on the capsules. It should also be noted that the laboratory equipment used in this work was not optimum or representative of available industrial units that would be more efficient for the intended application. Another approach to dry removal of the capsules might be some pretreatment of the workpiece surfaces to achieve release of the capsule.

The optimum capsule size or range of sizes for maximum "sensitivity" of the materials has not been determined. The very small capsules may enter a crack more readily than the larger capsules, but may be more difficult to remove because of higher surface energies. It may be more advantageous to have a large size distribution with a maximum upper size limit for some applications. If a smaller size microencapsulated penetrant were to be needed, such is available. Microcapsules of less than 1 micron diameter can be procured but only as a water slurry. At such small sizes it is not currently possible to furnish these in dry form. A more complete understanding of how the crack indication is formed with the capsules is required to decide upon capsule sizes.

The possible role and effectiveness of both dry and wet developers on crack image enhancement has not been clarified. In the testing that has been done, it does not appear that a developer provides a useful function if the part being inspected does not have shiny surfaces. The interrelationship between developers and capsule materials susceptible to attack or permeation by developer carriers has not been investigated in regard to improving crack indication visibility.

It is also apparent that the process using microencapsulated penetrant is readily automatable. One can envision an inspection line consisting of appropriate conveyors, parts holding fixtures, a spraying station which includes recovery and reuse of oversprayed capsules, a station at which excess capsules are removed by vacuum and brushing of the parts or by a water detergent wash, a drying station if water is employed, and then over to inspection under ultraviolet.

SECTION V

AREAS OF FURTHER WORK

Excellent promise of microencapsulated penetrants has been shown in the work reported herein. There are numerous areas of work that needs to be investigated.

The immediate need is to fully qualify this method of penetrant inspection and secure the confidence of the nondestructive community. This can be done by determining the sensitivity of the form of penetrant. In the tests reported herein it appears that the 2-5 micron size material will qualify as a Group VI, penetrant under MIL--25135. More tests on crack standards needs to be done to confirm this.

The defect detection efficiency needs to be determined in a typical production NDI line.

Spraying equipment and parameters need to be studied to arrive at the best application techniques. Removal of excess capsules on the surfaces by dry techniques require further investigation. This would include establishing the role of static charging, and eliminating static attraction, and more extensive investigation of vacuum and brushing. Wet removal techniques do not seem to require further work. The detergent water rinse appears to be very simple, rapid, and effective.

For these areas of further work the 2-5 micron and a larger size such as 8-10 micron microencapsulated penetrant should be employed. It is imperative that the microencapsulation be of high quality, nonporous, and washed to ensure avoidance of "leakers".

Other areas of interest but of lesser priority would include developing the lowest cost, simplest, brightest dye solution to be encapsulated, and studying microencapsulated penetrant in the form of a slurry rather than as dry capsules.

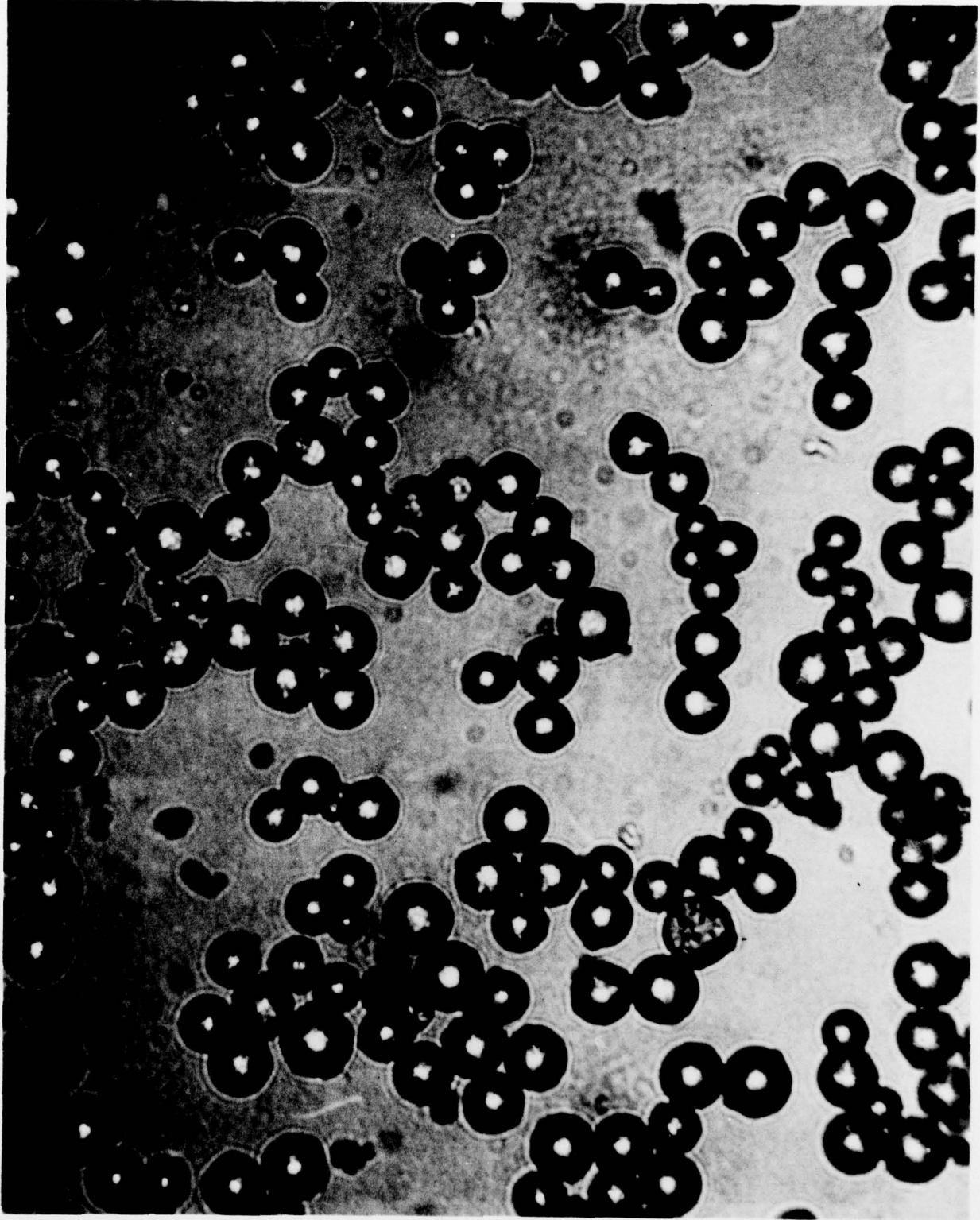


Figure 1. Microcapsules (8-16 microns)

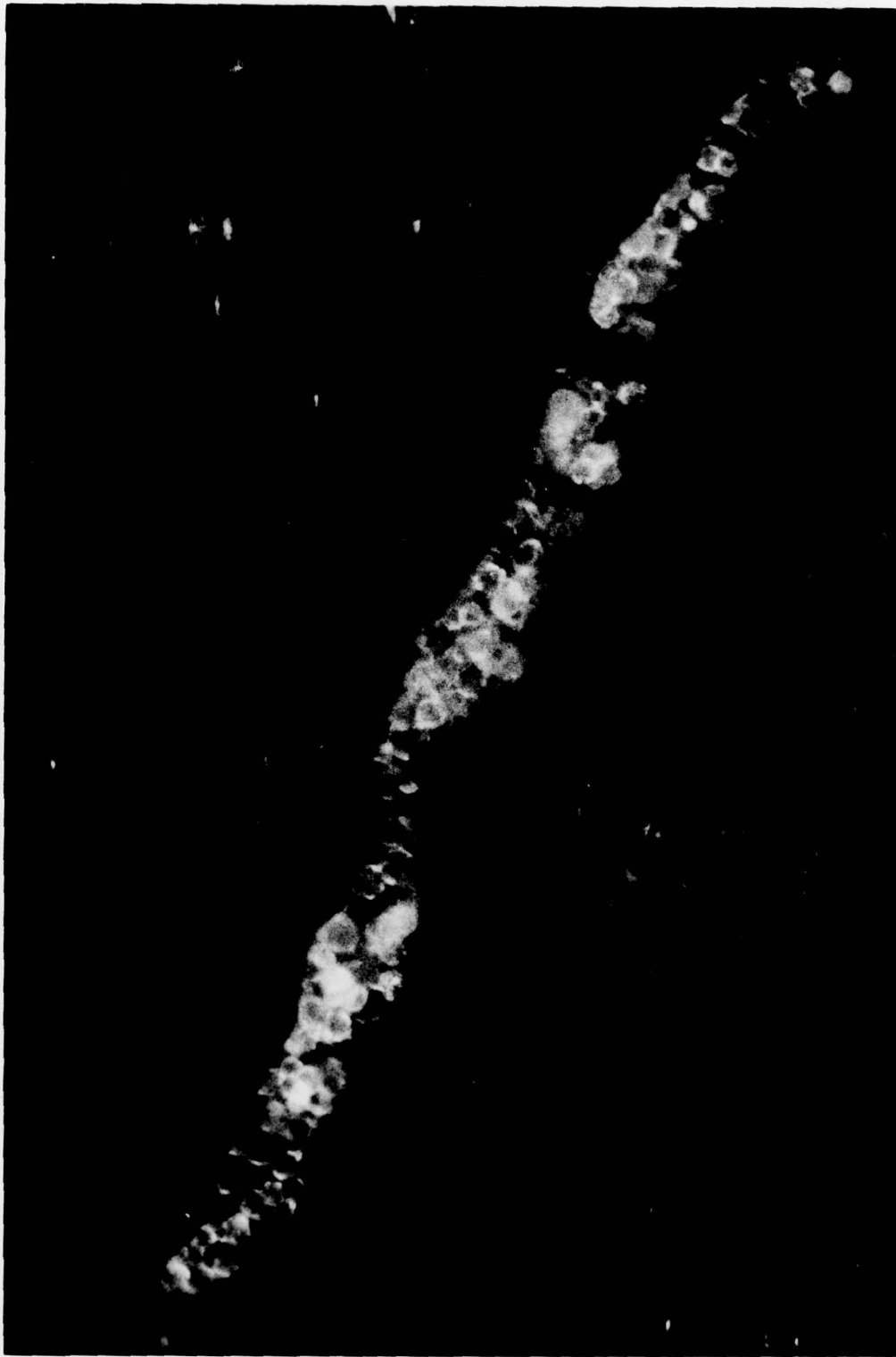


Figure 2. Microcapsules in Turbine Blade Crack

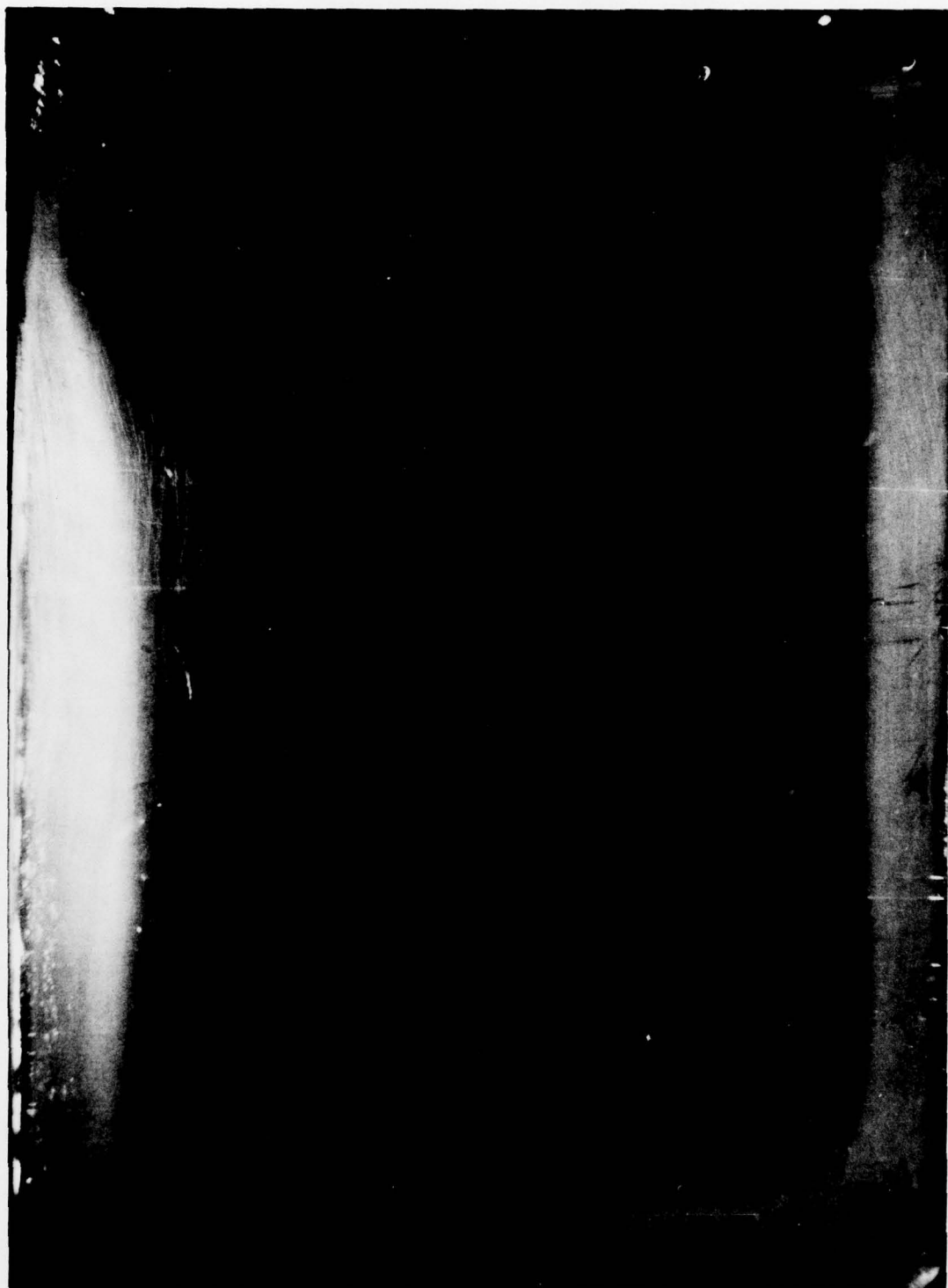


Figure 3. Monsanto (MRC) Fine Crack Standard



Figure 4. Steel Crack Standards

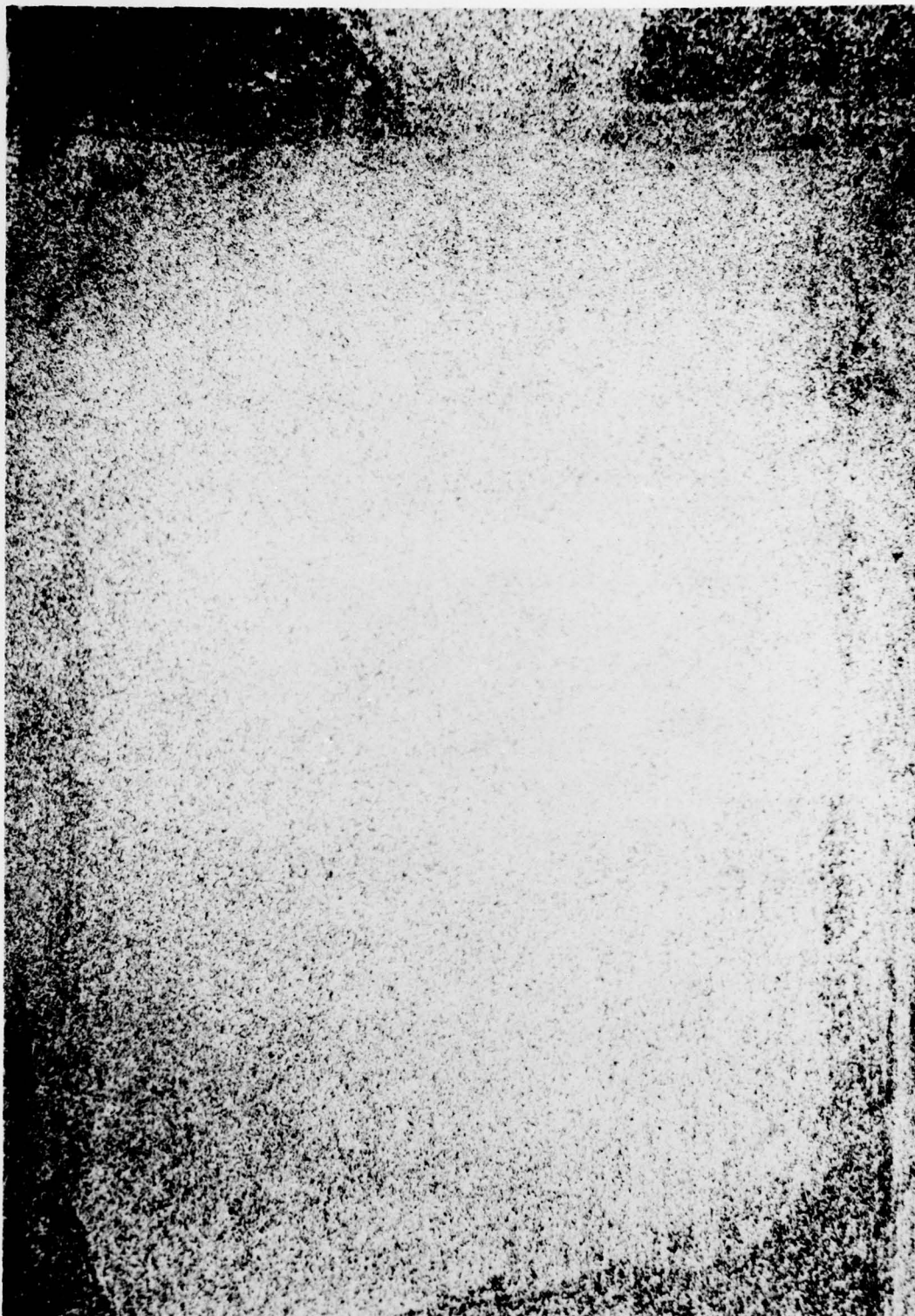


Figure 5. Capsule Applied by Conventional Spray Techniques



Figure 6. Capsules Applied by Air Eraser

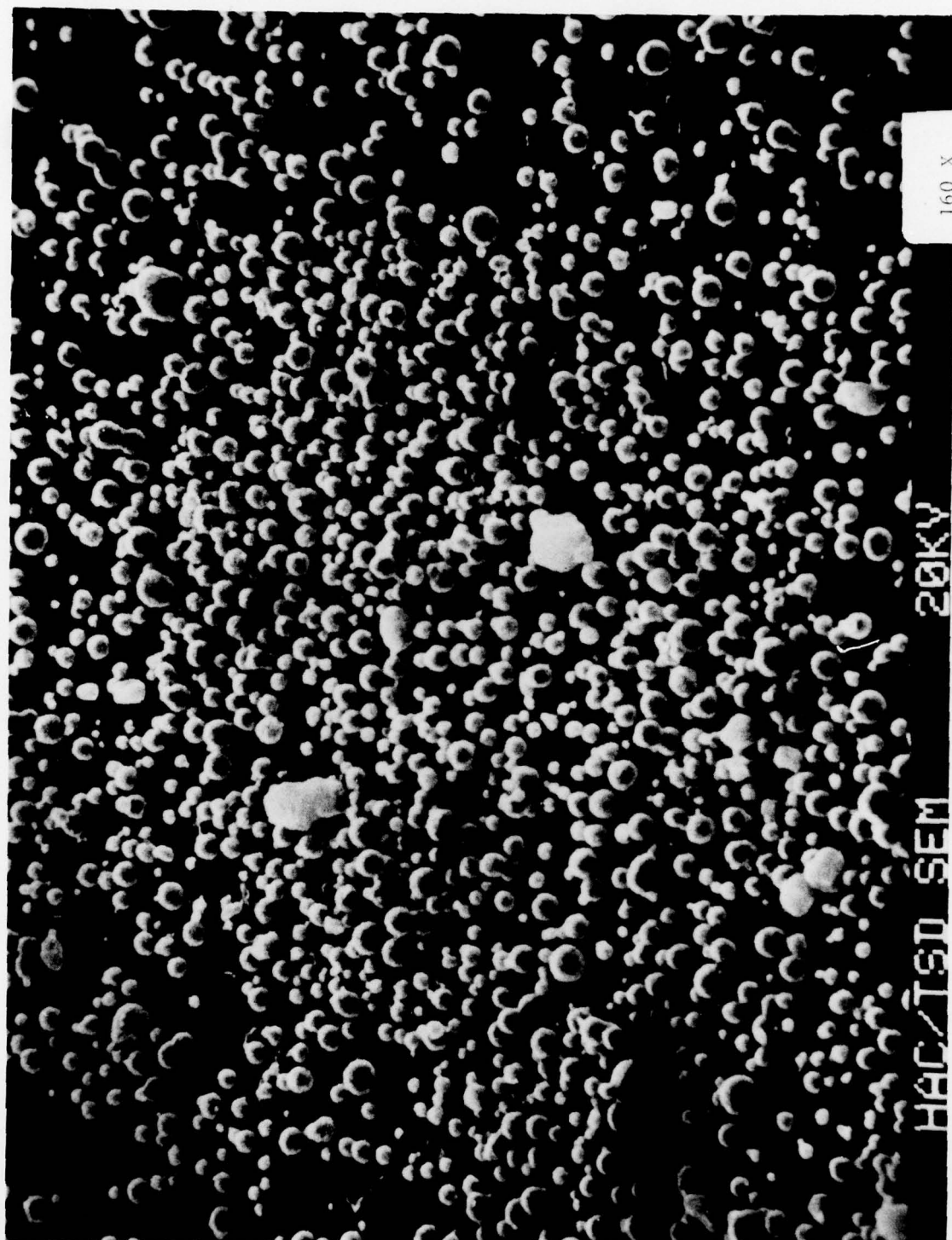


Figure 7. Scanning Electron Microscope Photograph of Microcapsule Agglomerates at 160 X Magnification

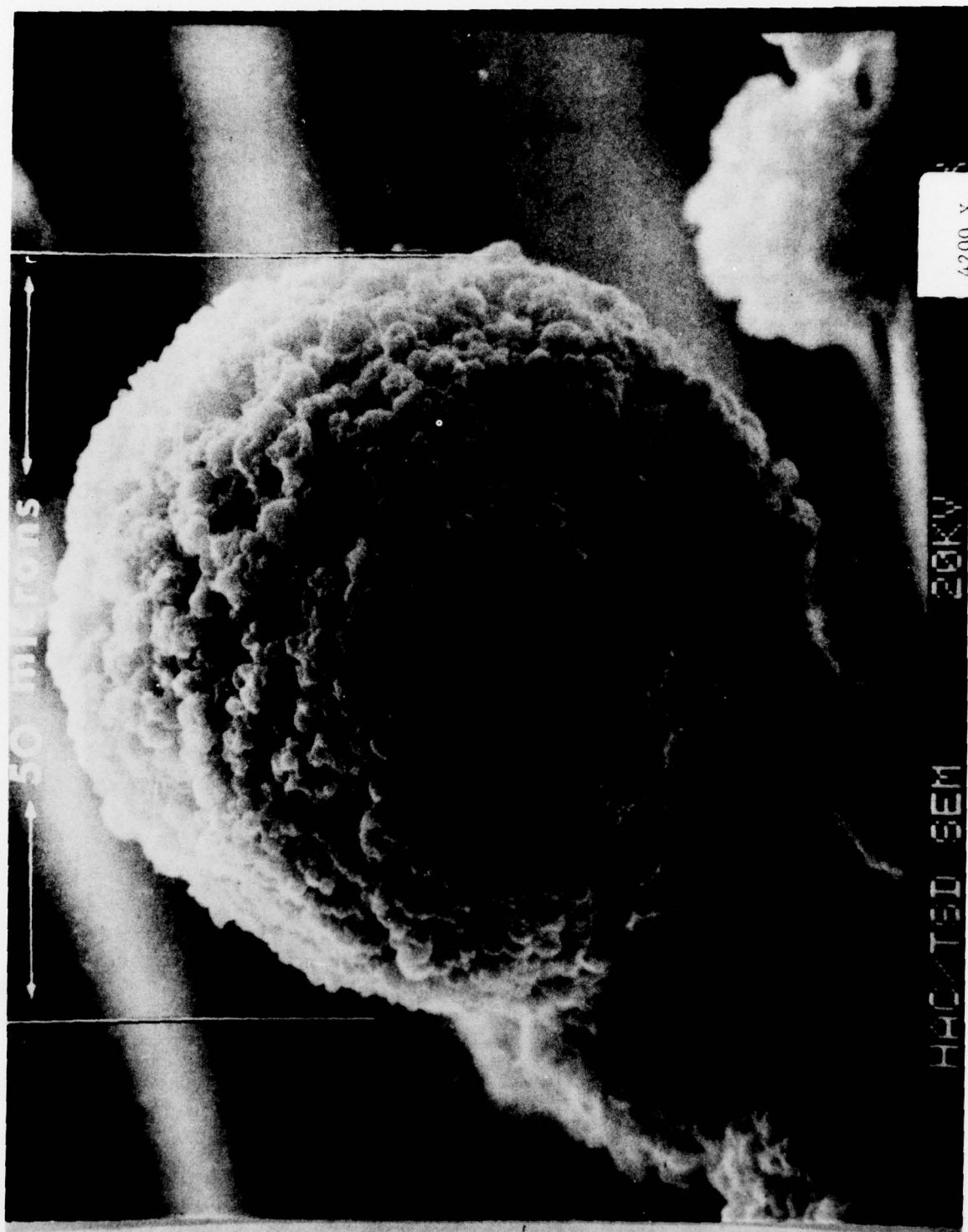


Figure 8. Scanning Electron Microscope Photograph of a Microcapsule Agglomerate at 4200 X Magnification

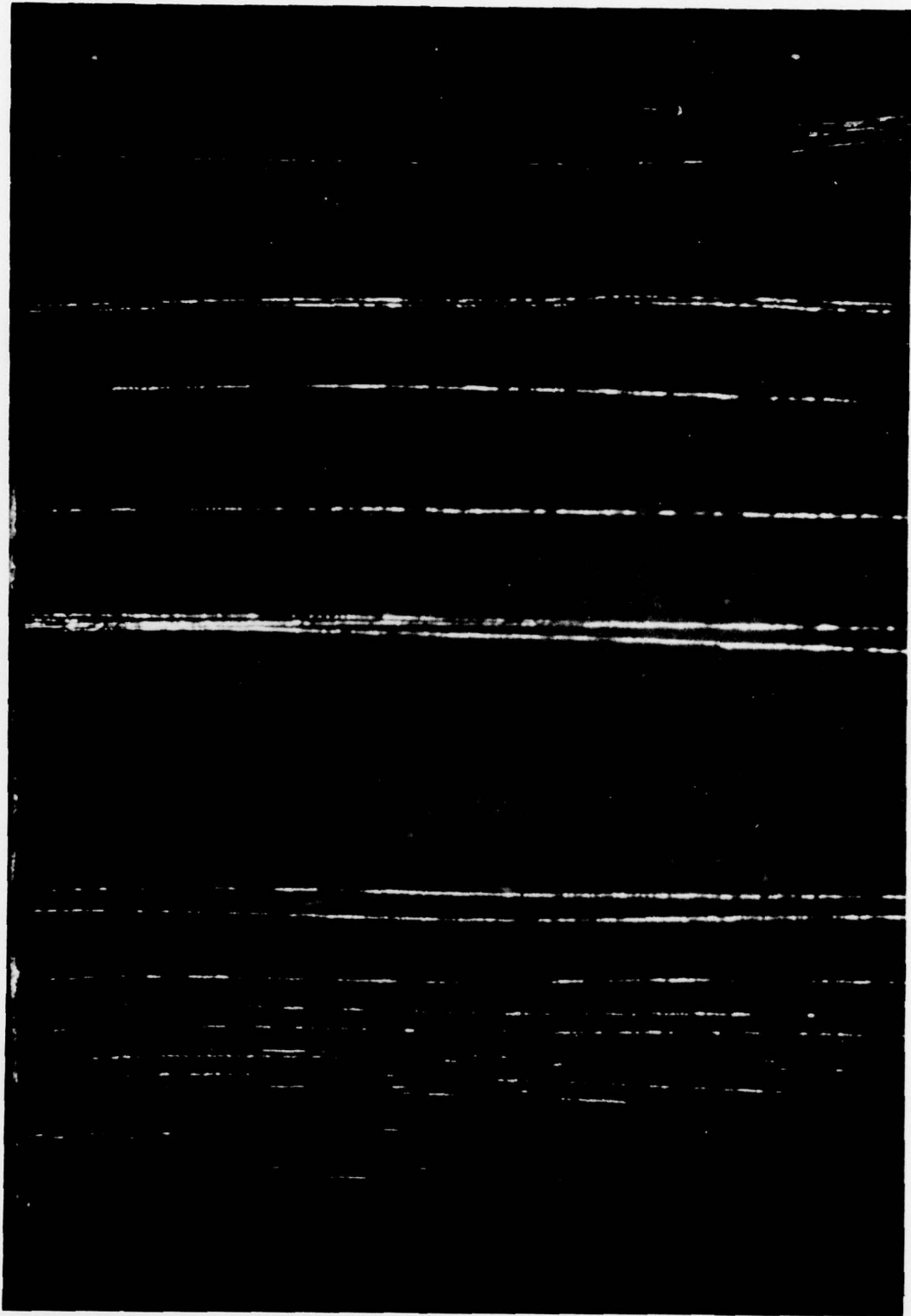


Figure 9. MRC Fine Crack Standard with Capsules in Cracks

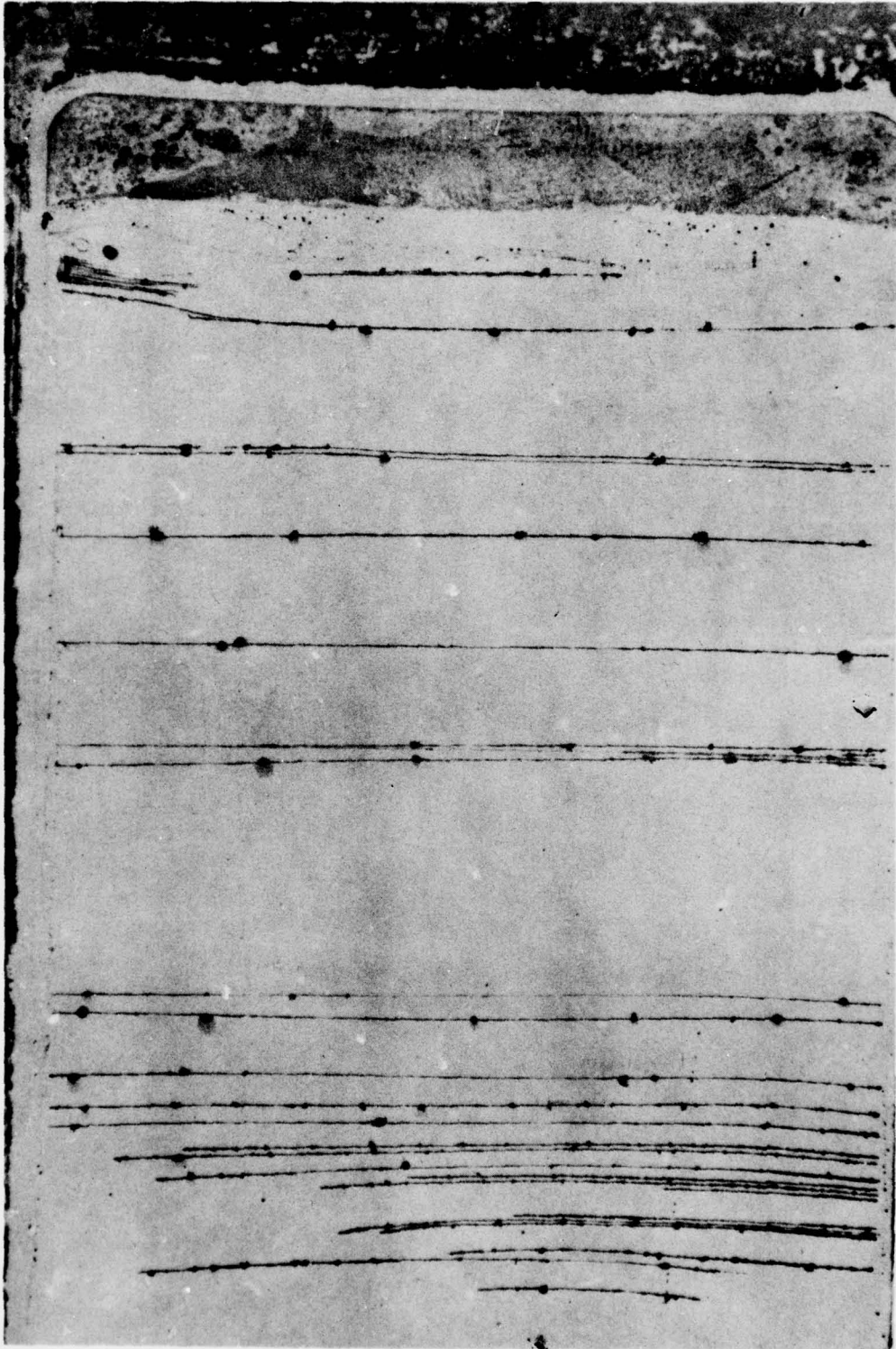


Figure 10. Electrographic Print of Figure 9 MRC Fine Crack Standard

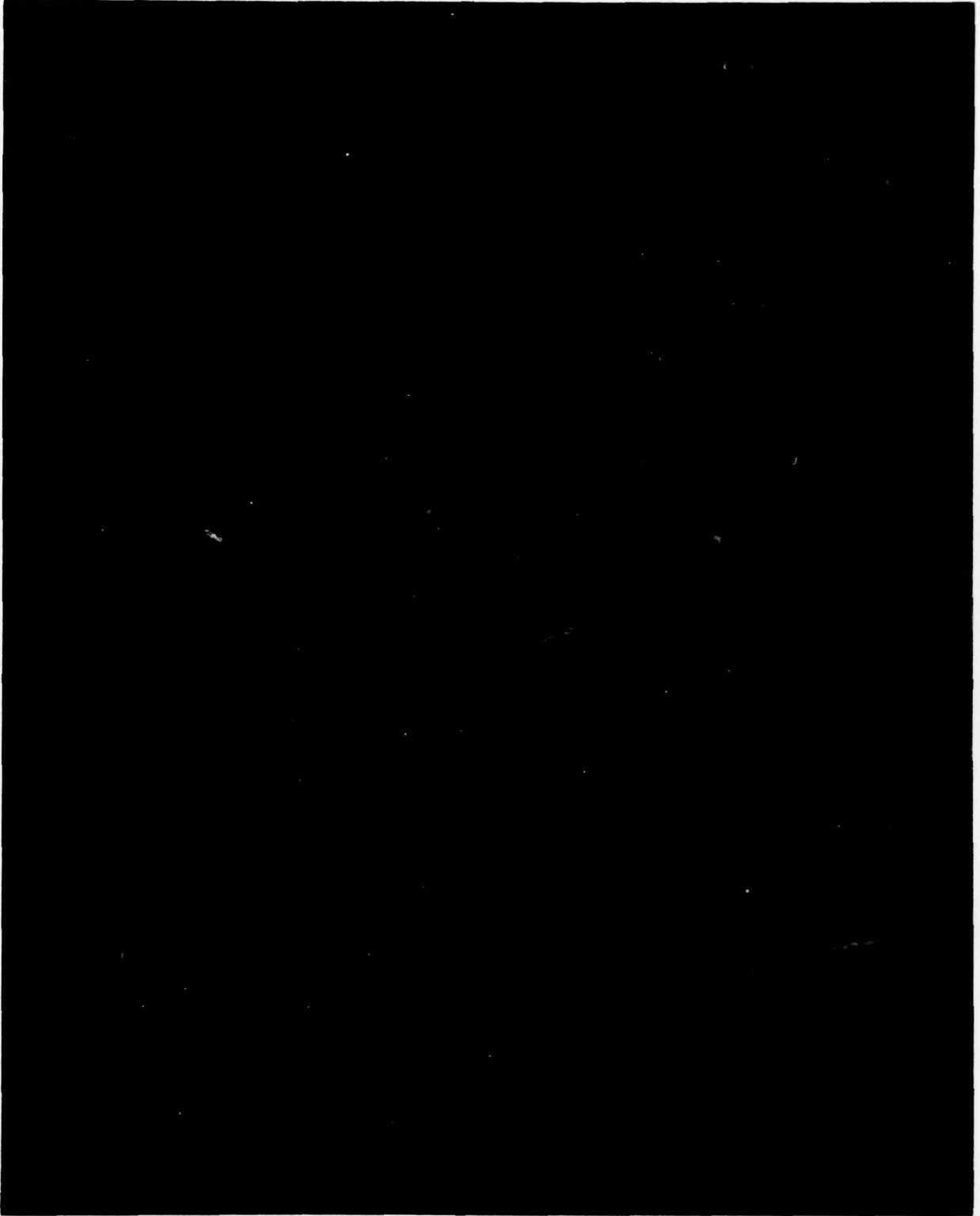


Figure 11. 2.5 Micron Width Fatigue Crack Indicated in Steel Crack Standard

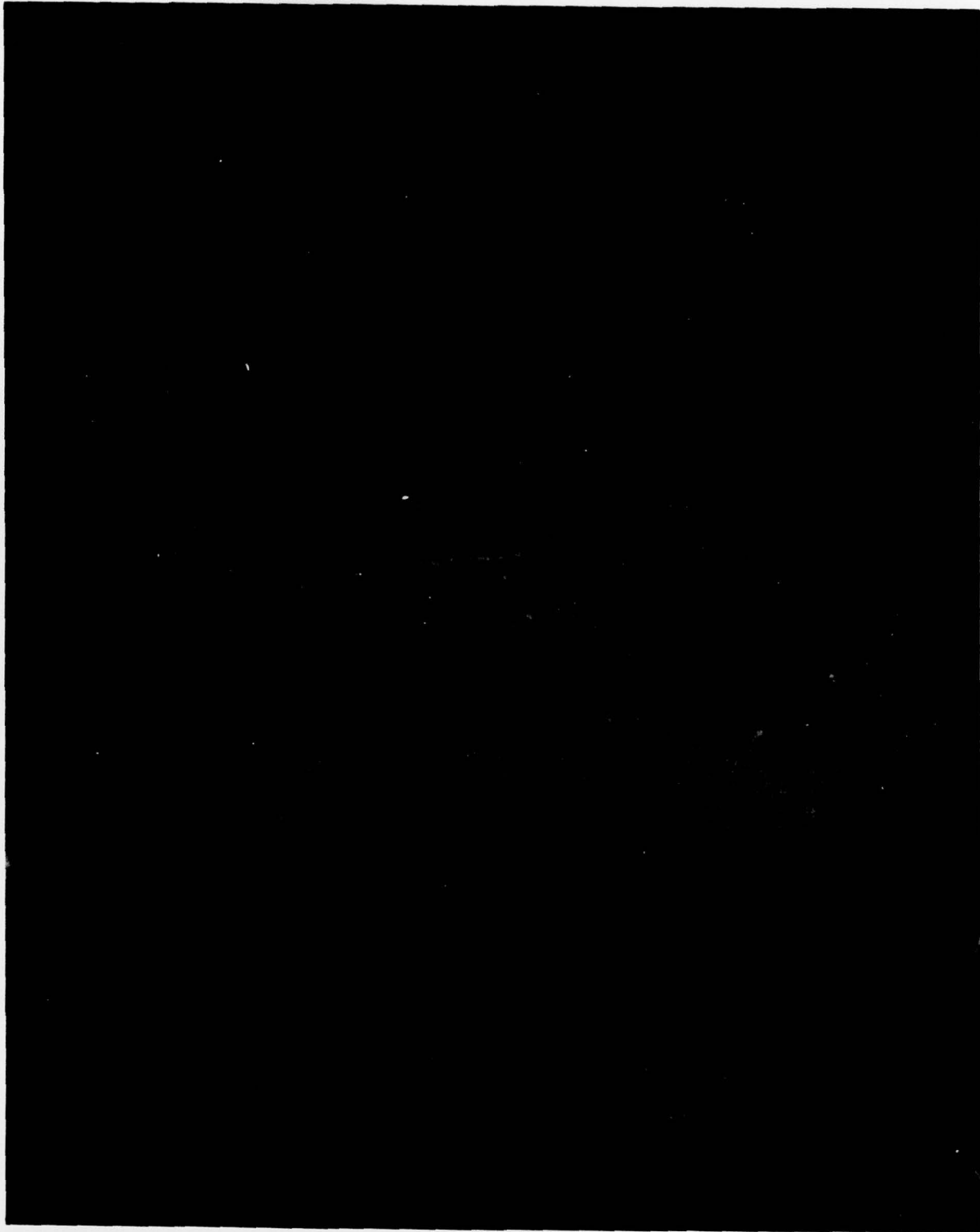


Figure 12. 0.5 Micron Width Fatigue Crack Indicated in Steel Crack Standard

REFERENCES

1. Richard L. Pasley, Liquid Penetrants, National Aeronautics and Space Administration NASA SP-5113, Nondestructive Testing, 1973, Pages 7-25.