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AN INTERIM REPORT ON THE MATERIALS AND SELECTION CRITERIA ANALYSIS FOR THE COMPACT IGNITION TOKAMAK TOROIDAL FIELD COIL TURN-TO-TURN INSULATION SYSTEM

Volume 2

PHOTOGRAPHIC SUMMARY OF LABORATORY TESTING - INTERIM RESULTS

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Date Published—January 1990

Prepared by the
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831-7294
operated by
MARTIN MARIETTA ENERGY SYSTEMS, INC.
for the
U.S. DEPARTMENT OF ENERGY
under contract DE-AC05-84OR21400

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ABSTRACT

This document contains photographs showing the results of laboratory testing of the combinations of epoxy resins, hardeners, and cures undertaken as part of the Compact Ignition Tokamak Insulation Screening Program. Cryogenic shock and soak to equilibrium proved to be the most demanding condition for these materials. The degree of damage to the basic materials when a poor candidate is selected is shown to be quite dramatic.

INTRODUCTION

This document contains photographs generated during the course of laboratory testing reported in ORNL/ATD-28, Vol. 1, *An Interim Report on the Materials and Selection Criteria Analysis for the Compact Ignition Tokamak Toroidal Field Coil Turn-to-Turn Insulation System.* Representative photographs were included in the first volume to illustrate specific points; however, the degree of difference in behavior of the various resin/hardener/cure combinations under cryogenic exposure was quite dramatic and warrants a more complete documentation of the sample condition.

Also included in this volume are representative photographs of the wet-out and compatibility samples. These tests did not disqualify any of the candidates directly but served to confirm flow problems with one system (Combination 7 – TGMDA analog). The photograph of this sample is included as the opposite of the typical sample.

LABORATORY SAMPLE SET

As stated in Vol. 1, the resin and hardener samples were prepared in several forms to provide test articles for several purposes. Resin and hardener were mixed at 45°C to produce the specimens for a given combination. The mix was deaerated and then used to produce the following samples.

NEAT RESIN AND EMBEDMENT SAMPLES

These blocks, ~2 1/2 in. in diam and ~0.50 in. thick, were poured in disposable aluminum pans. The neat-resin samples contain only resin. The embedment samples were produced by lightly scoring the bottom of the aluminum pan to hold a copper cylinder (1 in. in diam and 0.125 in. thick) slightly above the bottom of the pan. Figure 1 shows a set of samples for one combination of resin and hardener subjected to two different cure cycles.

WET-OUT AND COMPATIBILITY SAMPLES

Wet-out and compatibility samples were formed by placing the resin/hardener mix at 45°C on the materials of construction for the Compact Ignition Tokamak (CIT). The materials were glass cloth, Spaulrad S™ sheet material, copper sheet, and Inconel 718. Figure 2 shows fiberglass cloth tape and Spaulrad™ sheet samples covered by the small drop of resin/hardener mix applied. Note that the drop outline has been lost on the fiberglass tape. The drop has covered the Spaulrad™ sheet and run over the edge in some areas. Figure 3 shows droplet behavior on copper sheet, and Fig. 4 shows the droplet on Inconel. In both cases, the drop has wet the surface and is adherent. No incompatibilities (failure to cure, corrosive attack, or solvent attack) were observed.

COEFFICIENT-OF-THERMAL-EXPANSION SAMPLE

A small quantity of the resin/hardener mix was placed in a glass tube mold to produce a 0.25-in.-diam cylindrical sample for coefficient-of-thermal-expansion (CTE) measurement. Figure 5 shows the tube used as a mold, along with a sample that cracked itself due to thermal contraction during cool-down from cure. The sample was subsequently trimmed to length and the ends were finished to fit the test apparatus.

COMPRESSION TEST SAMPLES

Sheets of the resin/hardener combinations were cast and cut to produce 1-in. nominal square specimens (in thicknesses of 0.0125 and 0.625 in.) for testing.

PHOTOGRAPHS OF SIGNIFICANT RESULTS

The photographs that follow represent the significant results from the first series of laboratory evaluations. The majority of the photographs are backlit views of the neat resin and embedment resin blocks. Other items that warrant special notice are as follows.

1. These are photographs taken after ONE cryogenic shock/soak sequence.

2. All of the blocks that show a failure were prompt failures. Snapping and crackling indicative of failure began immediately on immersion. None of the samples cracked while returning to ambient or later at room temperature.

3. The photographs are marked with the two-number coding that designates the resin/hardener combination and the cure cycle. For more detailed descriptions, see Vol. 1 of this report.

   Combinations 1-1 and 1-2 (Figs. 6 through 9) - Baseline system bisphenol A resin with nadic methyl anhydride (NMA).

   Combinations 2-1 and 2-2 (Figs. 10 through 13) - Baseline system bisphenol A resin with flexibilized amine curing agent. This combination is one that did not fail.

   Combination 3-2 (Figs. 14 and 15) - Represents the system used for the ATF/STX insulation system. It is a bisphenol A with NMA and flexibilized anhydride hardener. Only one cure cycle, the constrained cycle, is defined for this system.

   Combinations 4-1 and 4-2 (Figs. 16 through 19) - Bisphenol F resin system. Lower viscosity and potential better cryogenic tolerance warranted inclusion of this system.

Combinations 5–1 and 5–2 (Figs. 20 through 25) – This combination used a bisphenol A resin flexibilized with a reactive ether and NMA as the hardener. Figure 20 shows the effects of a very aggressive hardener combined with a high-temperature cure. Portions of the neat resin block spalled off after cure (without cryogenic exposure). Cryogenic shock/soak did no further damage. Reduced volume copper embedment compared with the volume of the resin was not a significant driver in performance.

Combination 6 – was intended to be a bisphenol A/NMA system using an inert filler to help tailor CTE for a closer match with metals. The fillers could not be obtained in time to test the combination for this report.

Combinations 7–1 and 7–2 (Figs. 26 through 30) – A mixture of bisphenol A resin with methylene dianiline was used to produce a system that was the analog of tetraglycidylmethylene dianiline (TGMDA; also known as TGDDM). This is a known highly radiation resistant resin system. While the system did not shatter, it did show a stress crack (in the form of a conical shear surface) at the higher property cure (Fig. 27). The wet-out samples that are shown in Fig. 30 highlight the high viscosity of the system. Even after several hours at temperatures above 400°F, the outline of the drop on the cloth was still very evident. Flow at 45°C was very slight.

Combinations 8–1 and 8–2 (Figs. 31 through 34) – This system is a mixture of bisphenol A resin with cycloaliphatic amine hardener. This system has very desirable flow properties. The system survived the cryogenic test in good condition for one cure despite not having any flexibilizers added. This means that further tailoring of handling and performance can be undertaken, if needed.

INTERIM CONCLUSION

From the laboratory cryogenic testing, only three combinations of resin, hardener, and cure cycle survived. Of these, one (the TGMDA analog) was not amenable to use because of high viscosity and very rapid rise in viscosity. The surviving two candidates are (1) the heavily flexibilized bisphenol A resin/flexibilized amine system and (2) the bisphenol A resin with cycloaliphatic amine hardener.

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(U)

Fig. 1. Typical neat-resin and embedment sample blocks

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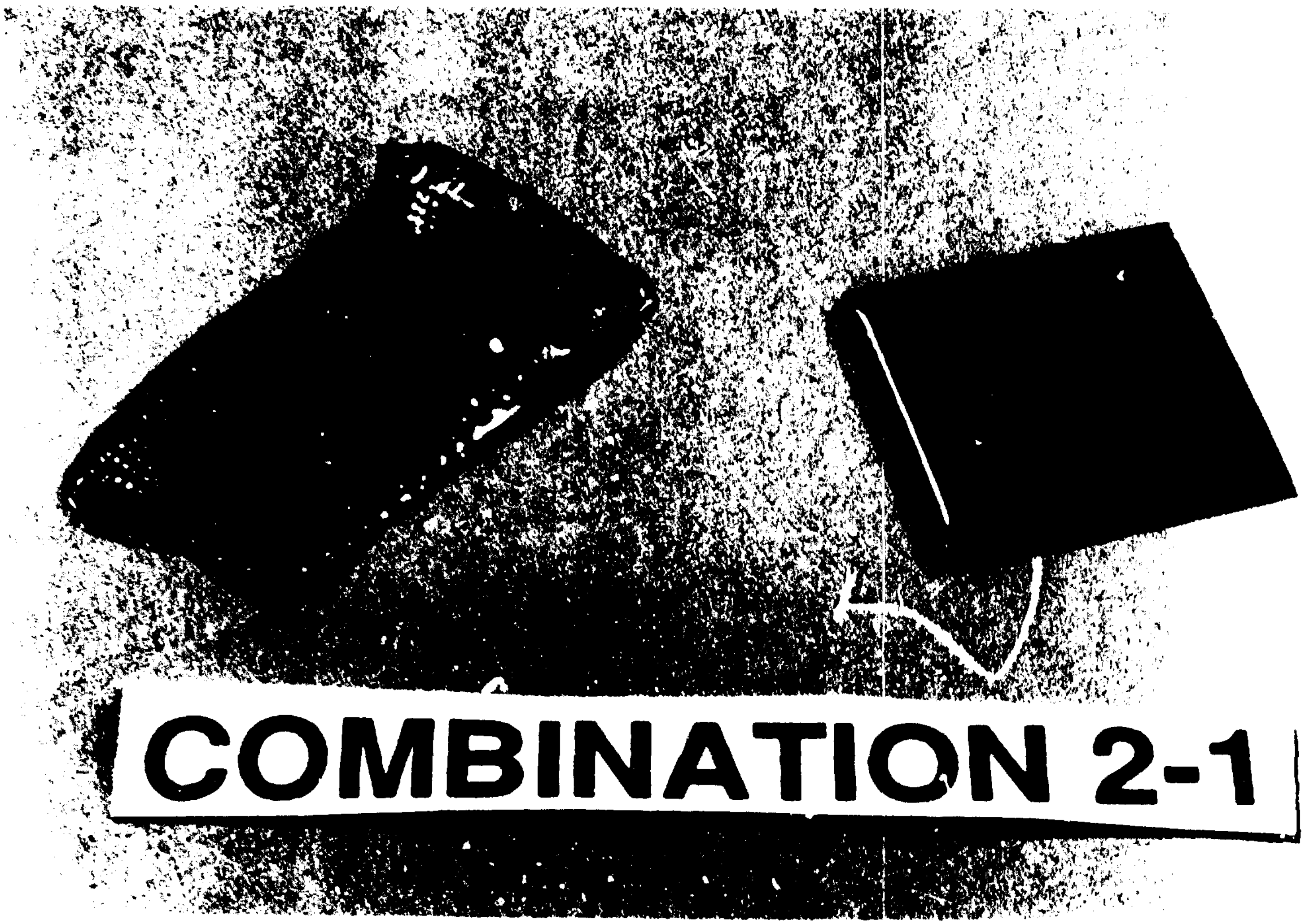

Fig. 2. Typical wet-out and compatibility samples

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(U)

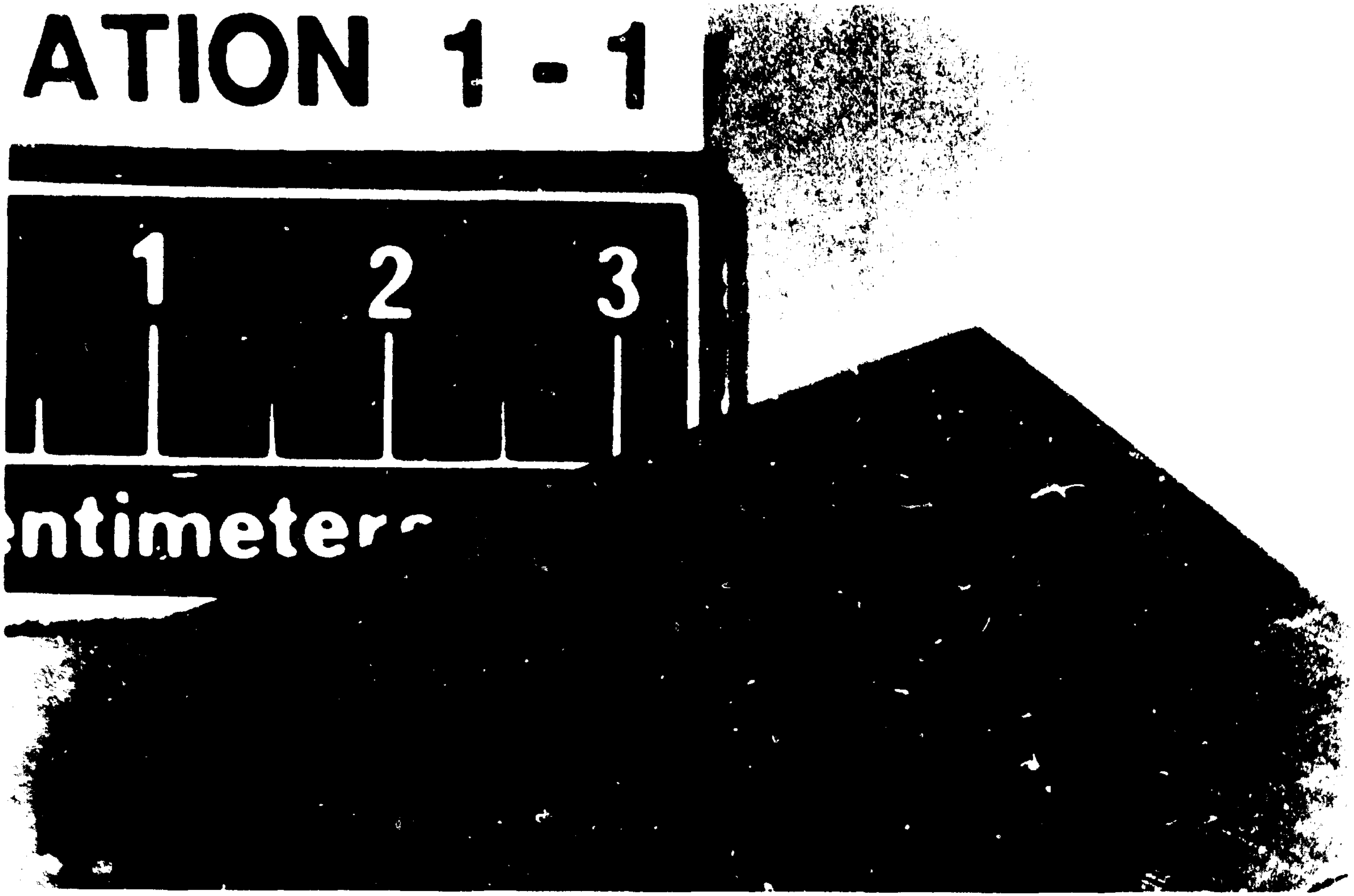

Fig. 3. Wet-out sample on copper substrate

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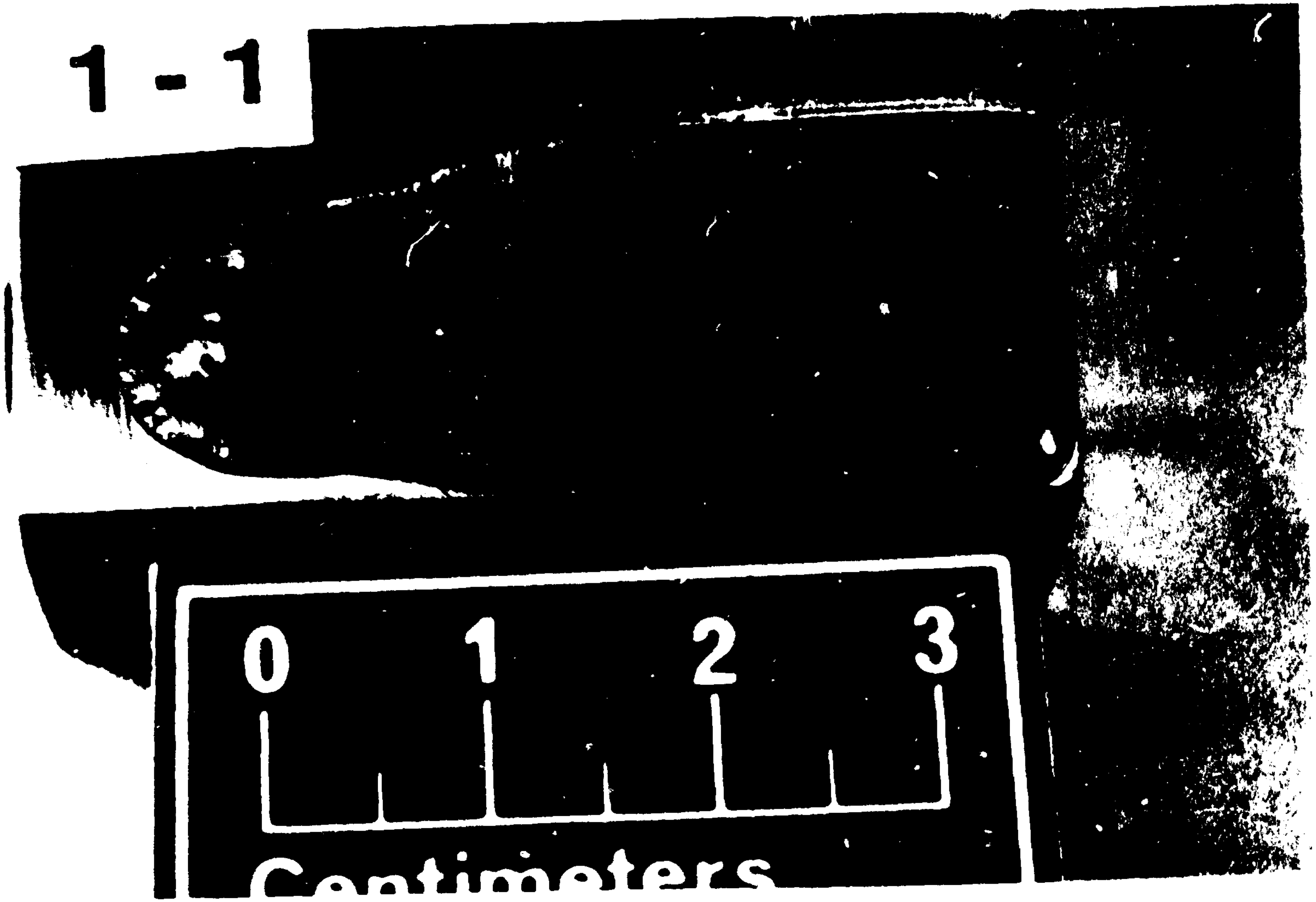

Fig. 4. Wet-out sample on Inconel substrate.

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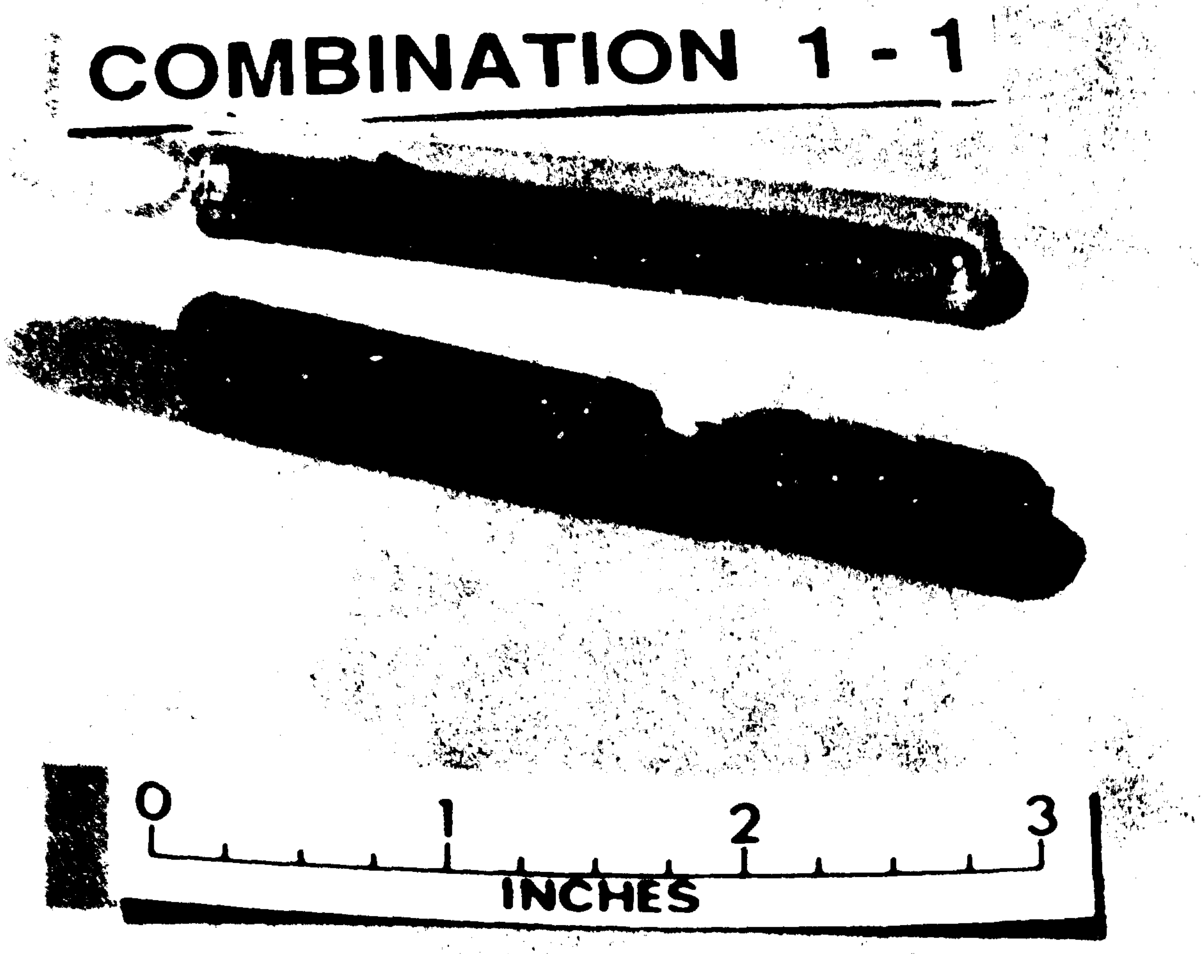

Fig. 5. CTE sample mold and sample cracked from thermal stress.

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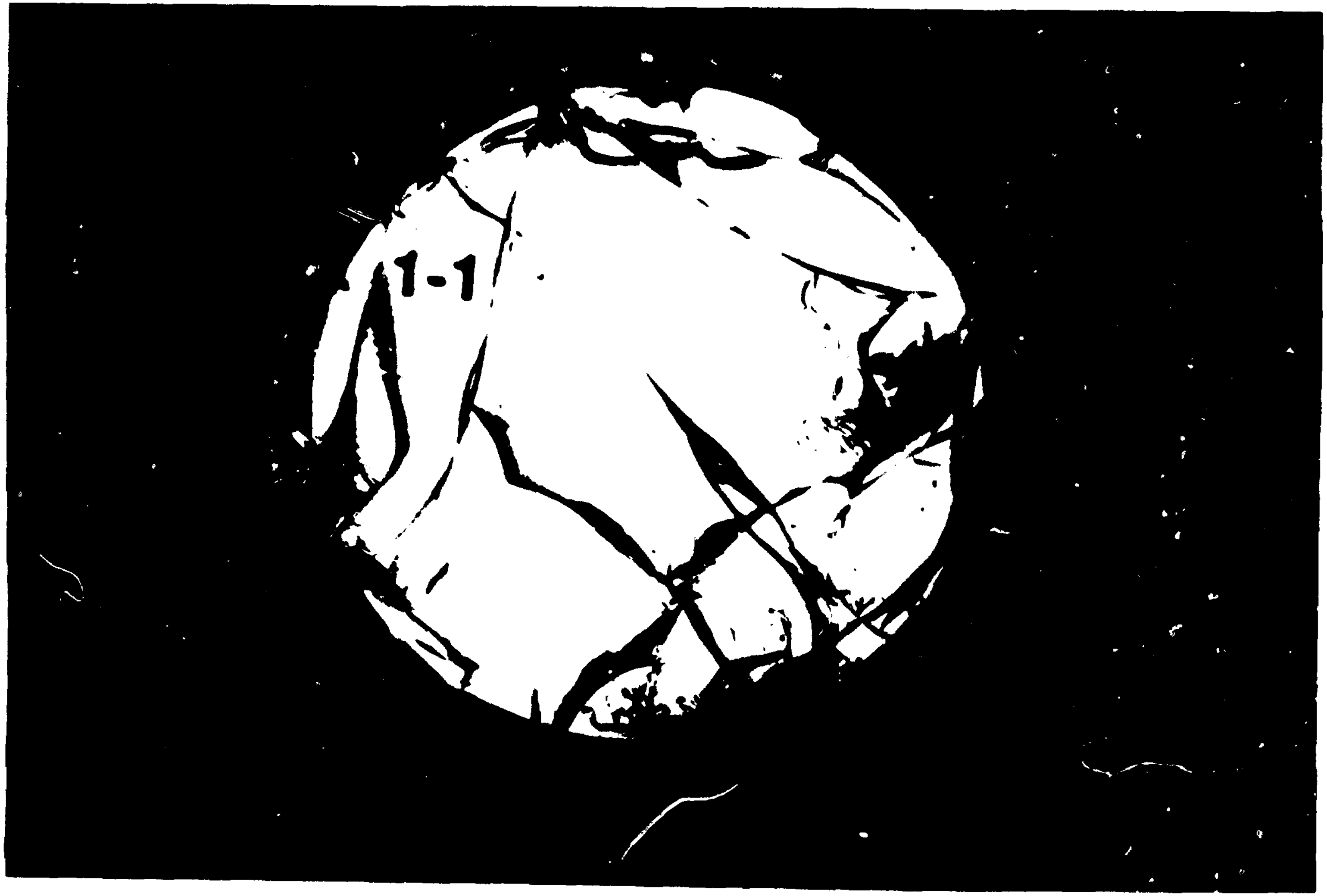

Fig. 6. Combination 1-1 neat-resin sample after cryogenic shock/soak

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(U)

Fig. 7. Combination 1-1 embedment sample after cryogenic shock/soak

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(U)

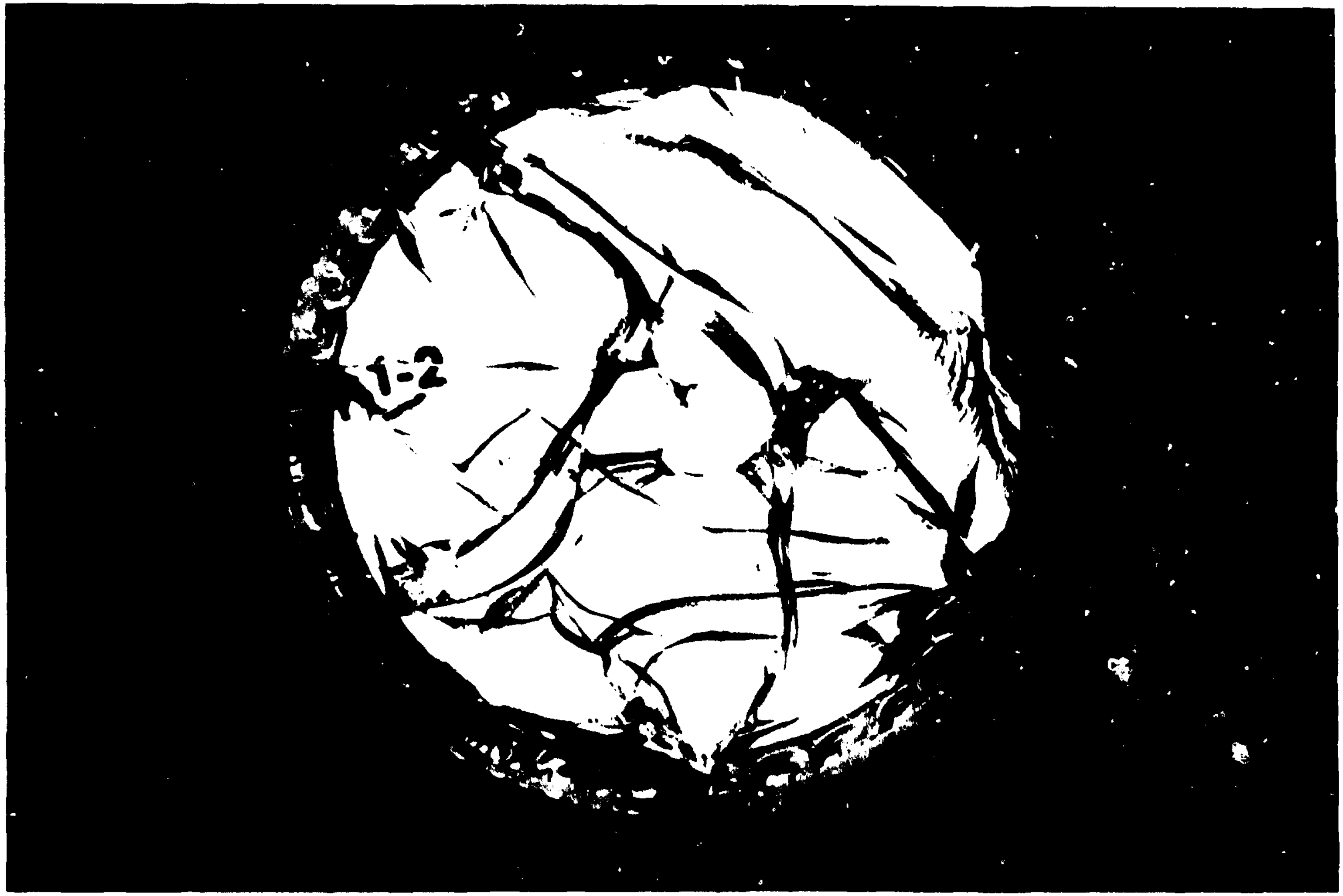

Fig. 8. Combination 1-2 neat-resin sample after cryogenic shock/soak

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(U)

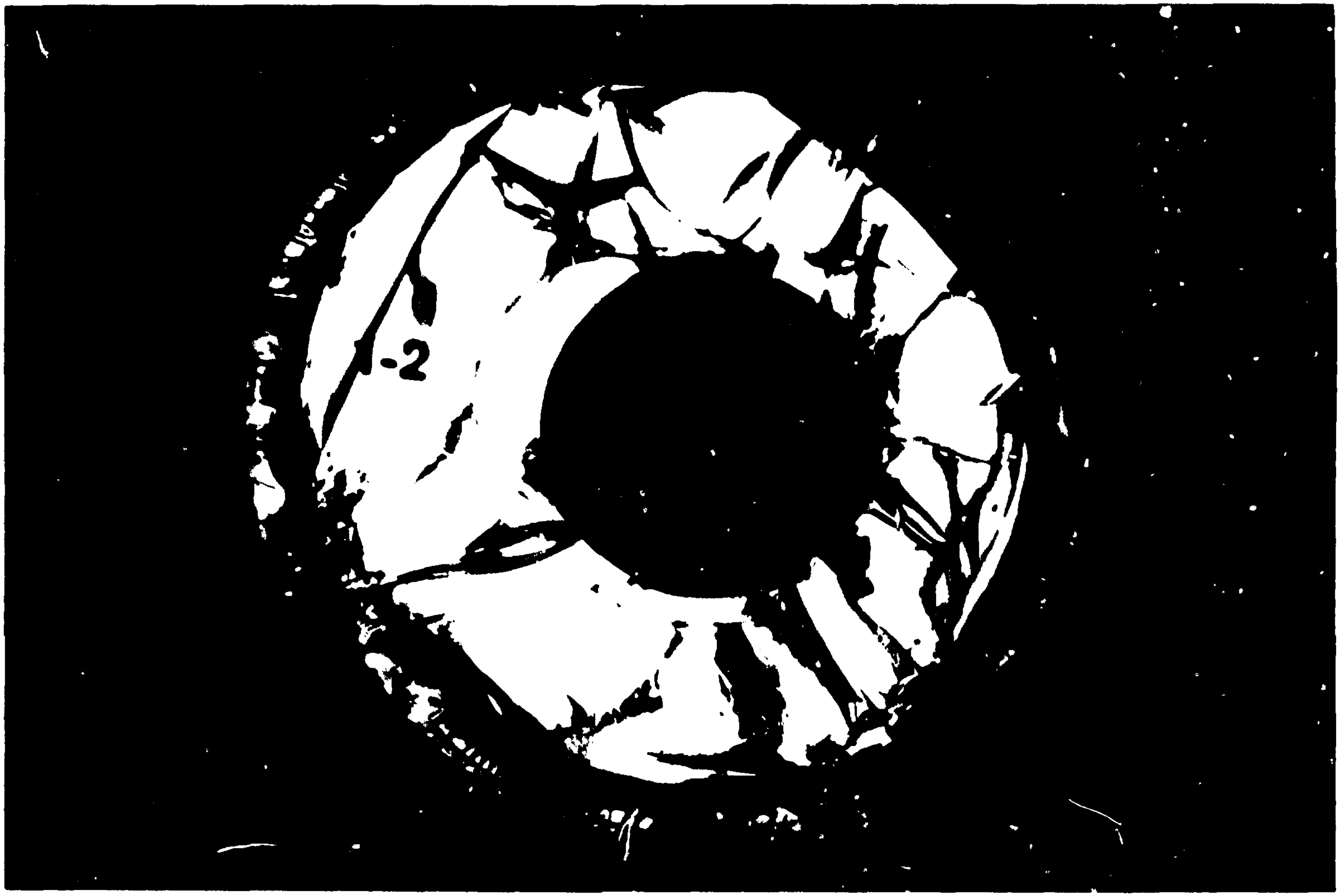

Fig. 9. Combination 1-2 embedment sample after cryogenic shock/soak

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Fig. 10. Combination 2-1 neat-resin sample after cryogenic shock/soak

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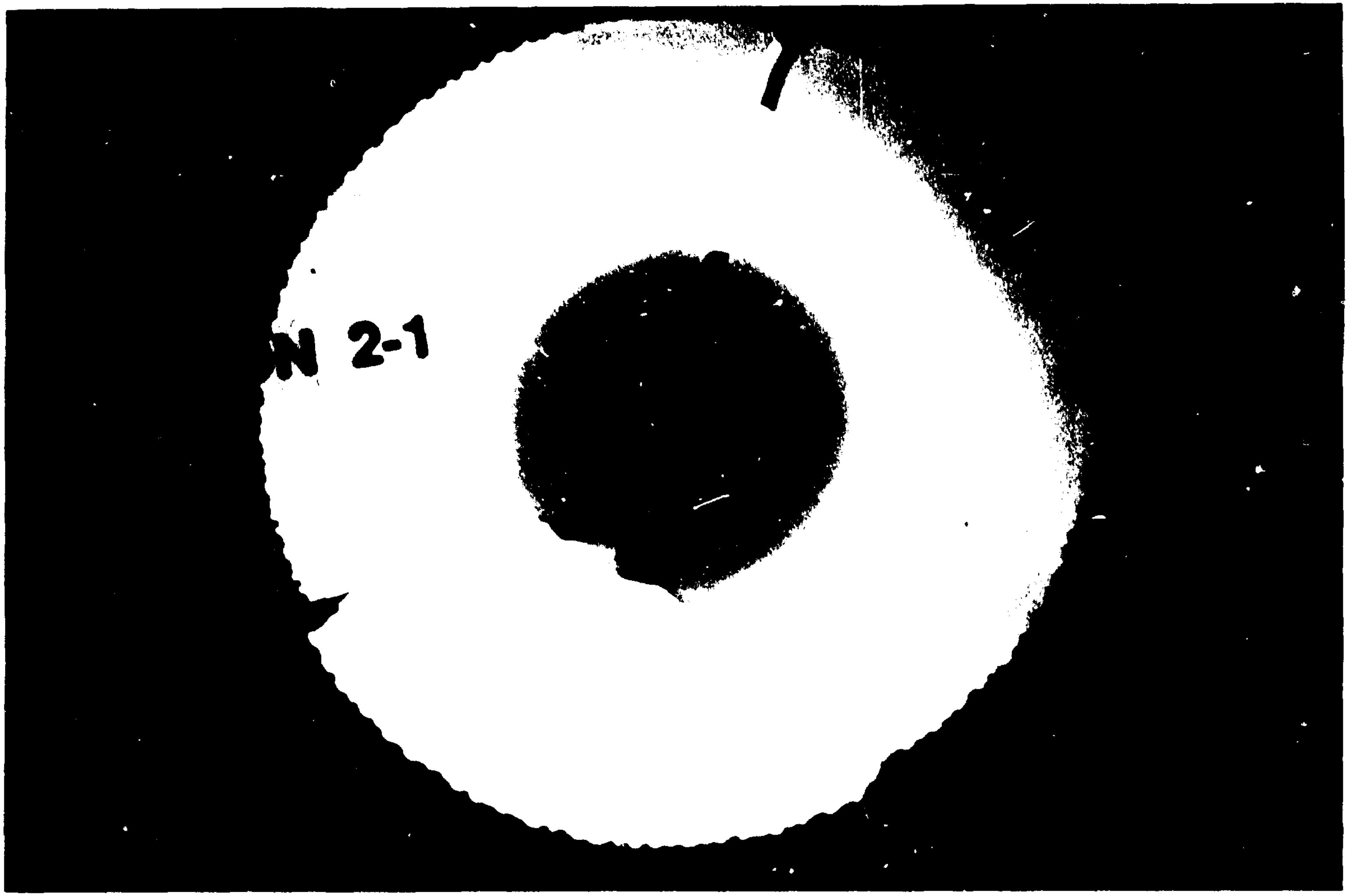

Fig. 11. Combination 2-1 embedment sample after cryogenic shock/soak

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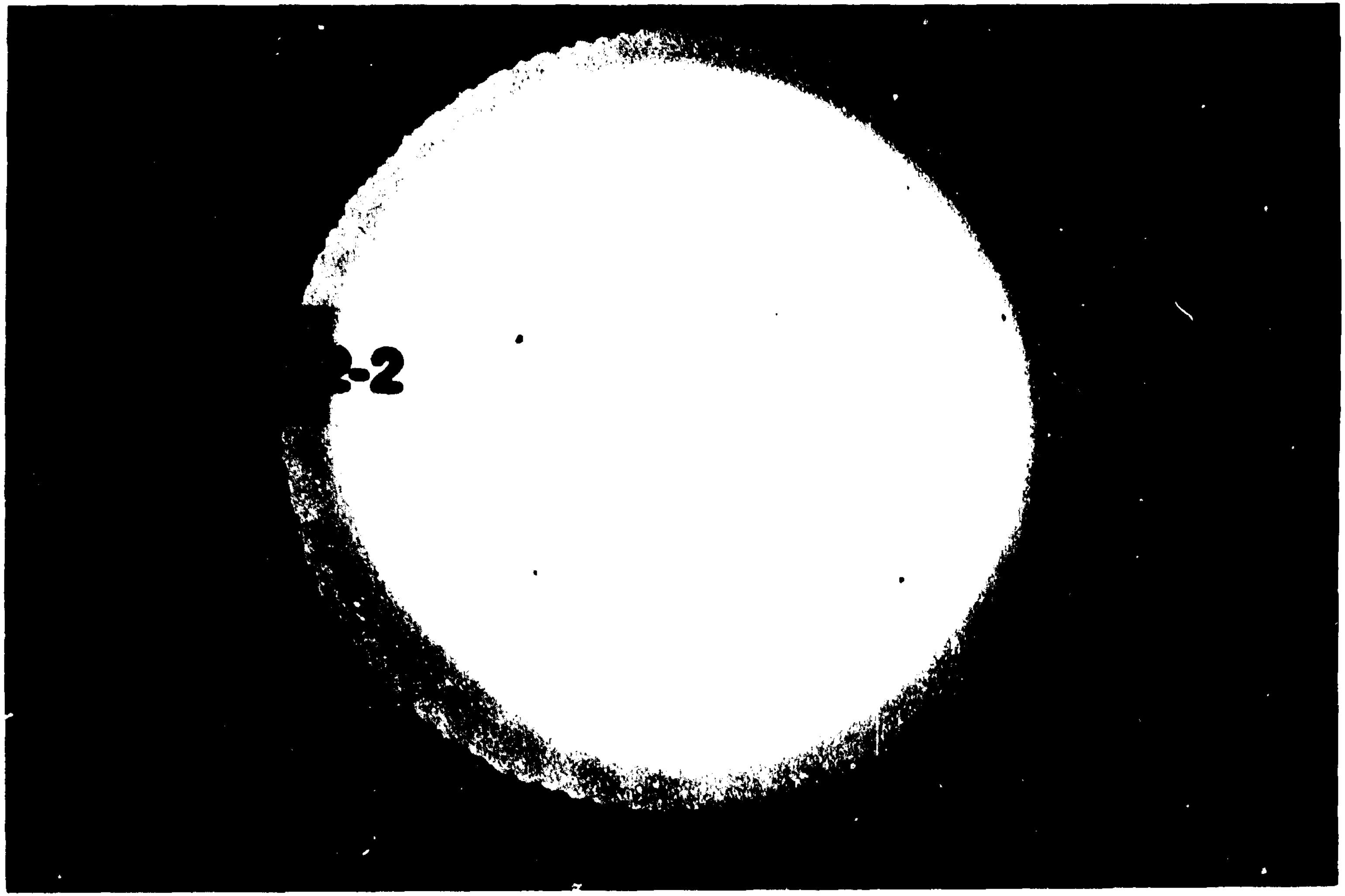

Fig. 12. Combination 2-2 neat-resin sample after cryogenic shock/soak

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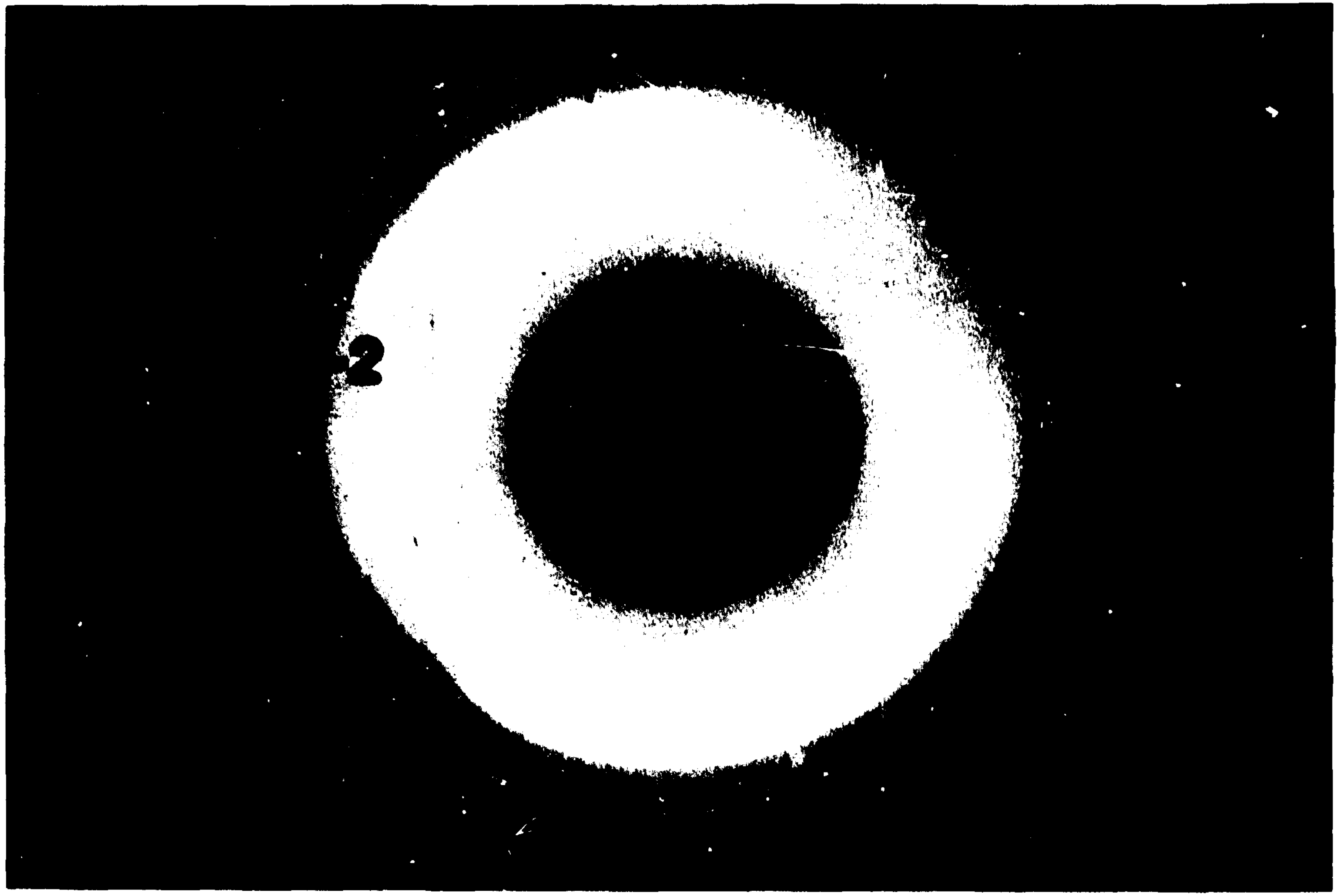

Fig. 13. Combination 2-2 embedment sample after cryogenic shock/soak

PHOTO NO. K/PH-89-2260
(U)

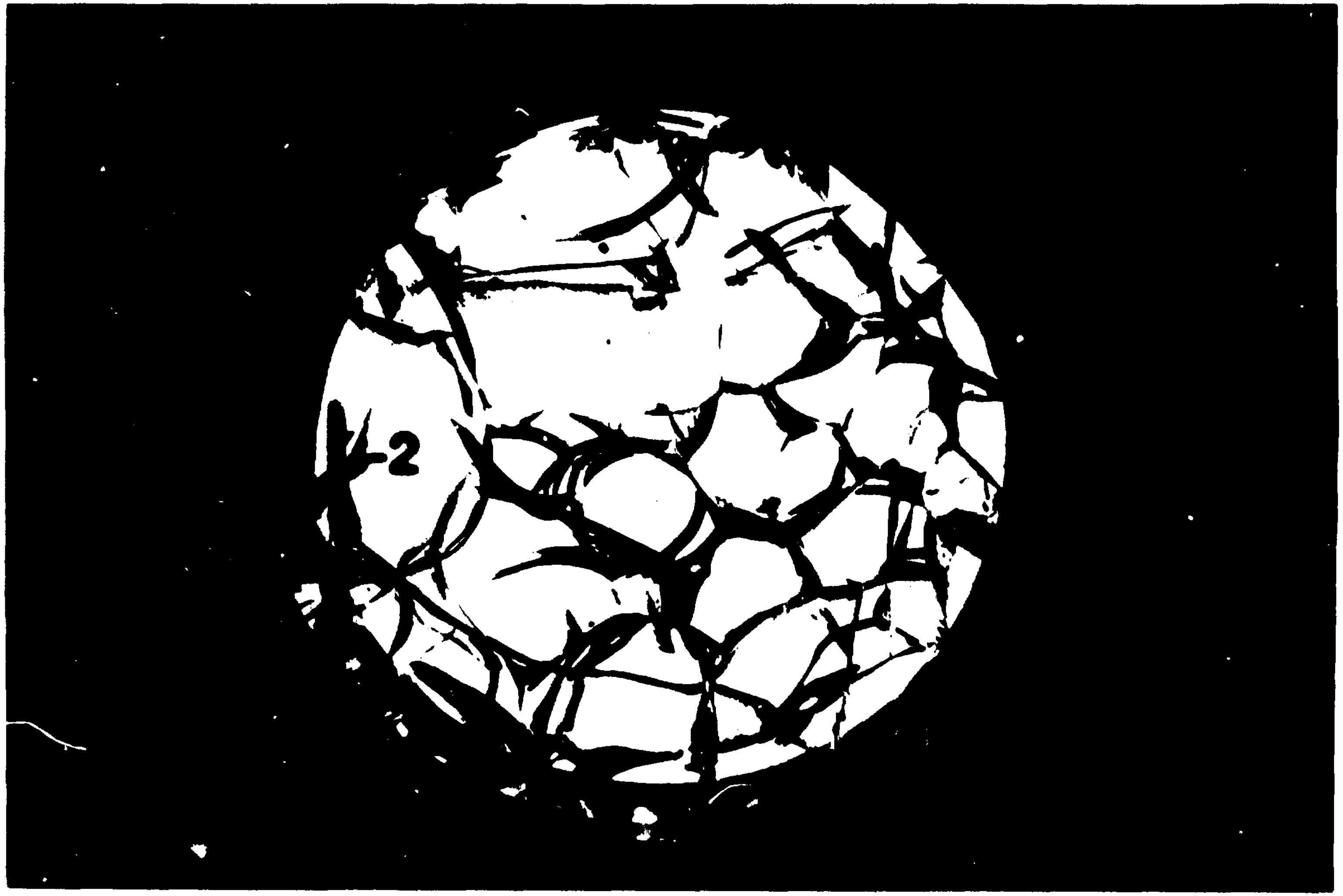

Fig. 14. Combination 3-2 neat-resin sample after cryogenic shock/soak

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(U)

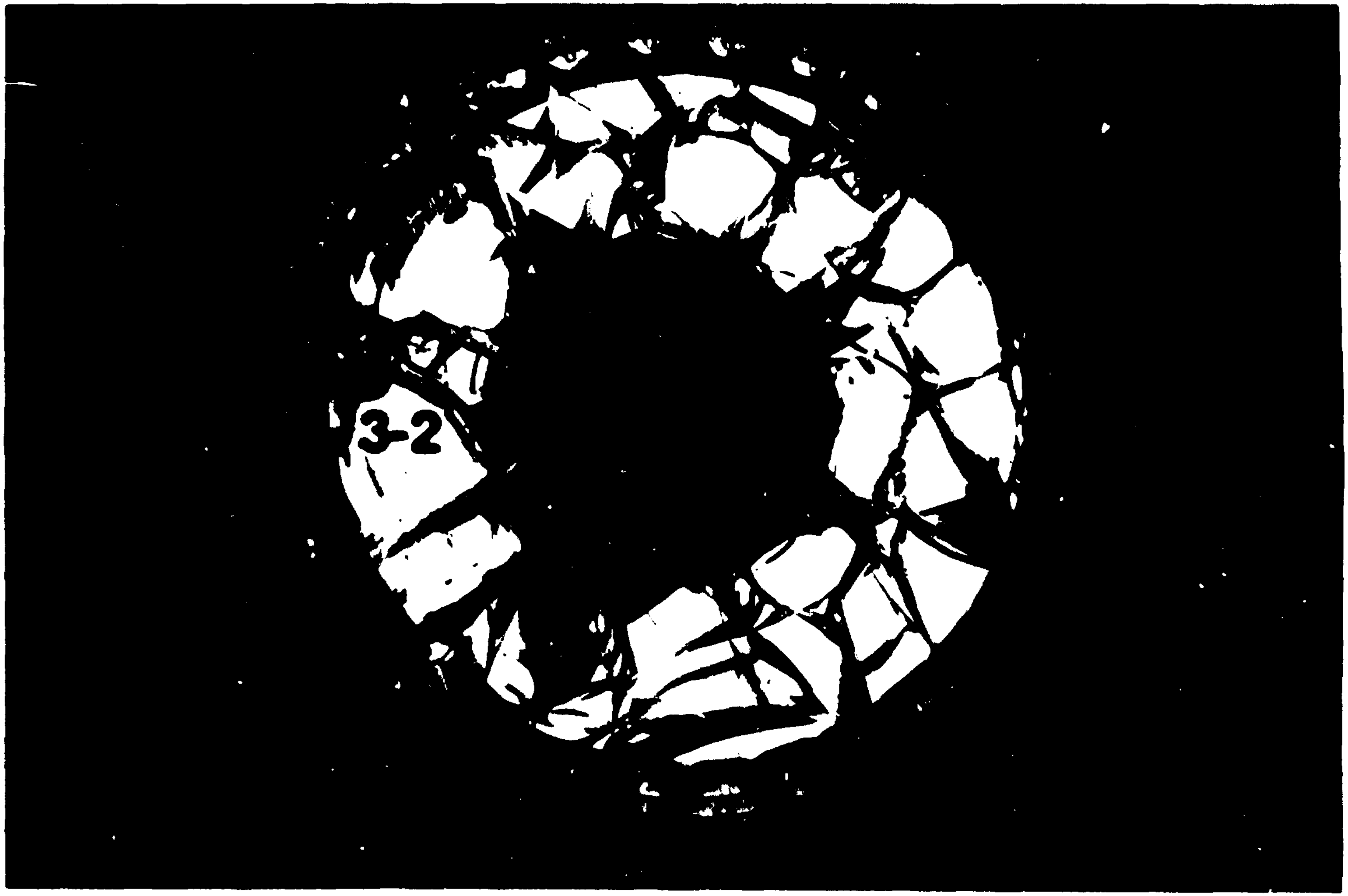

Fig. 15. Combination 3-2 embedment sample after cryogenic shock/soak

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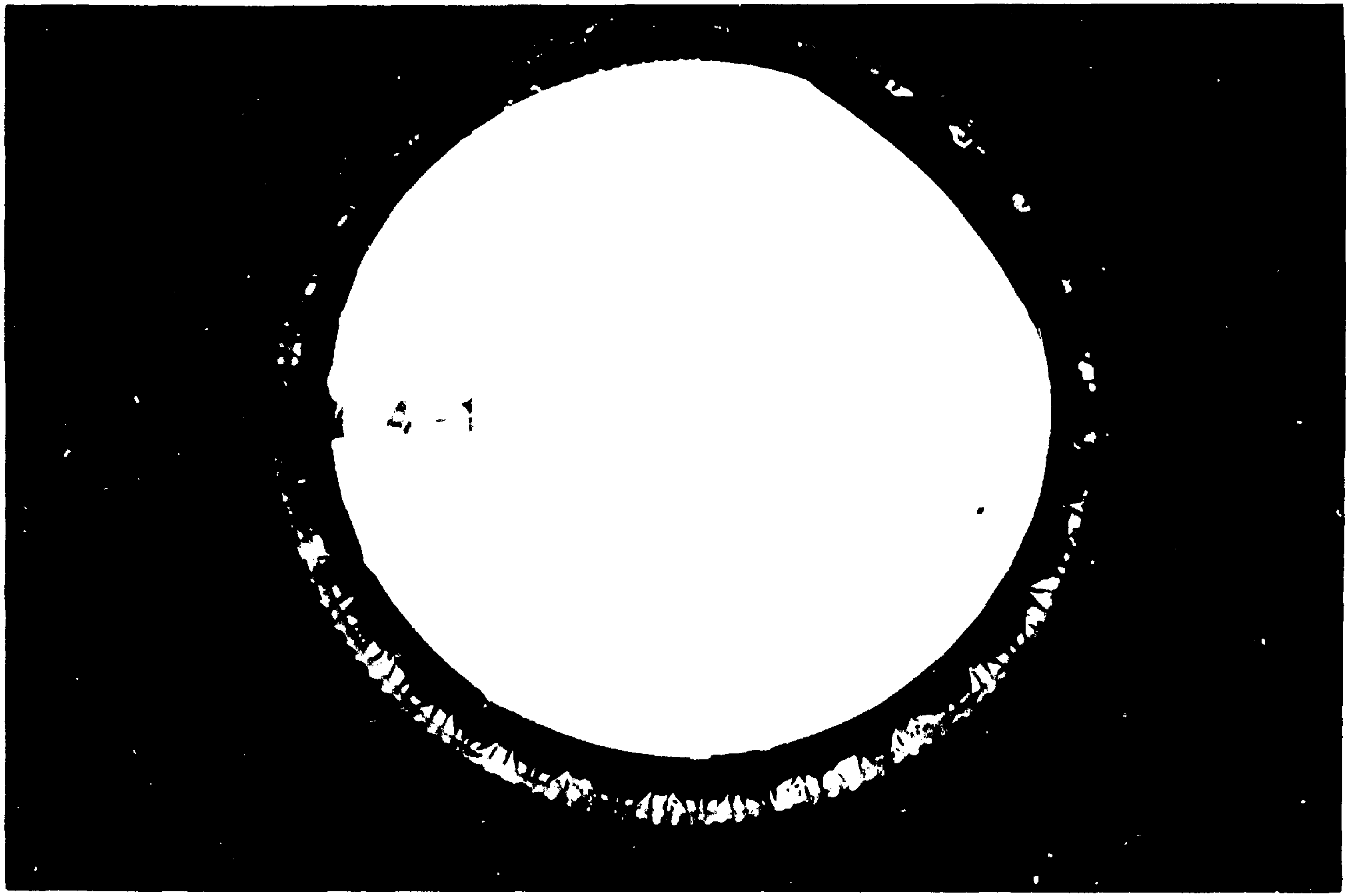

Fig. 16. Combination 4-1 neat-resin sample after cryogenic shock/soak

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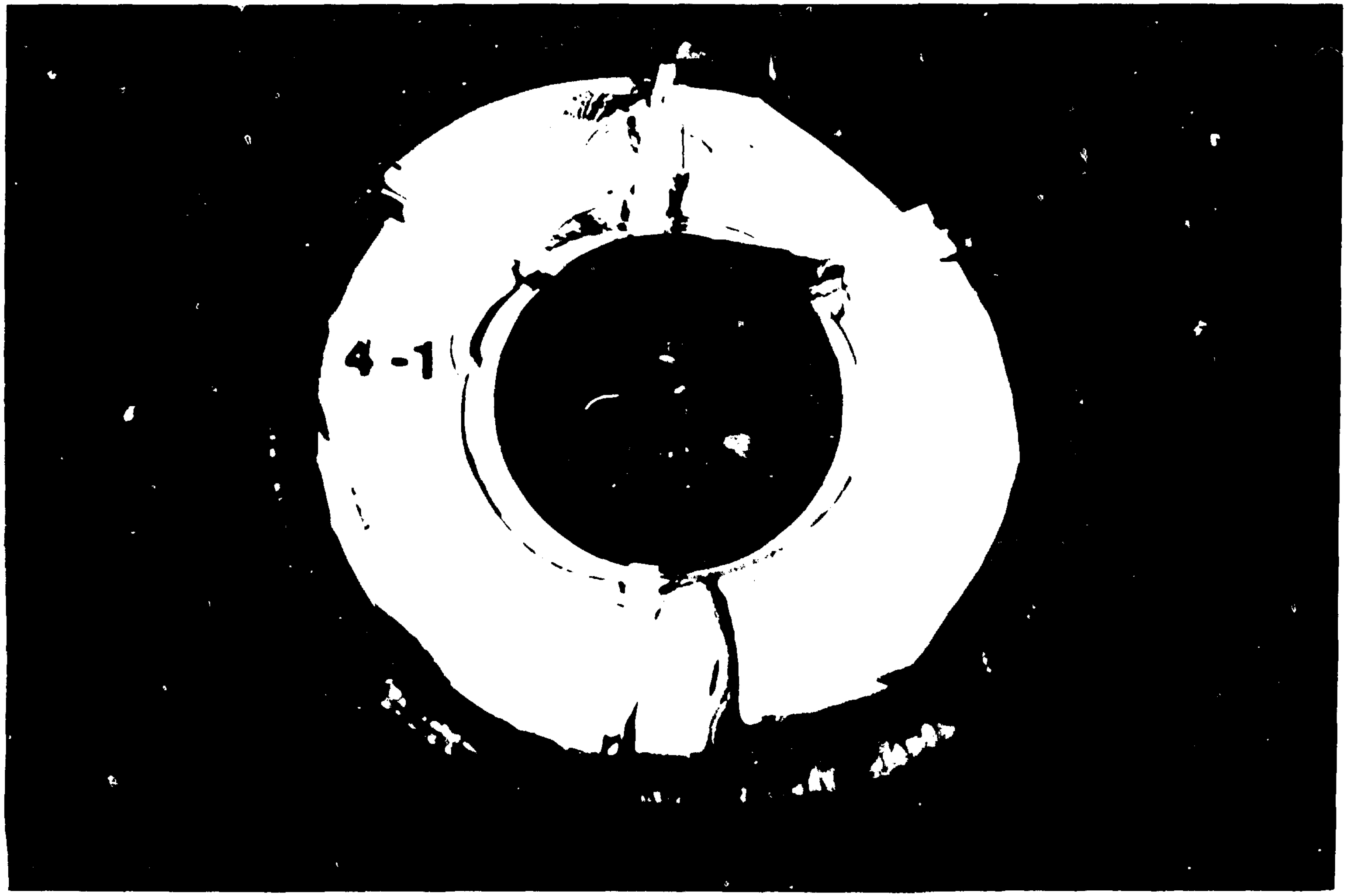

Fig. 17. Combination 4-1 embedment sample after cryogenic shock/soak

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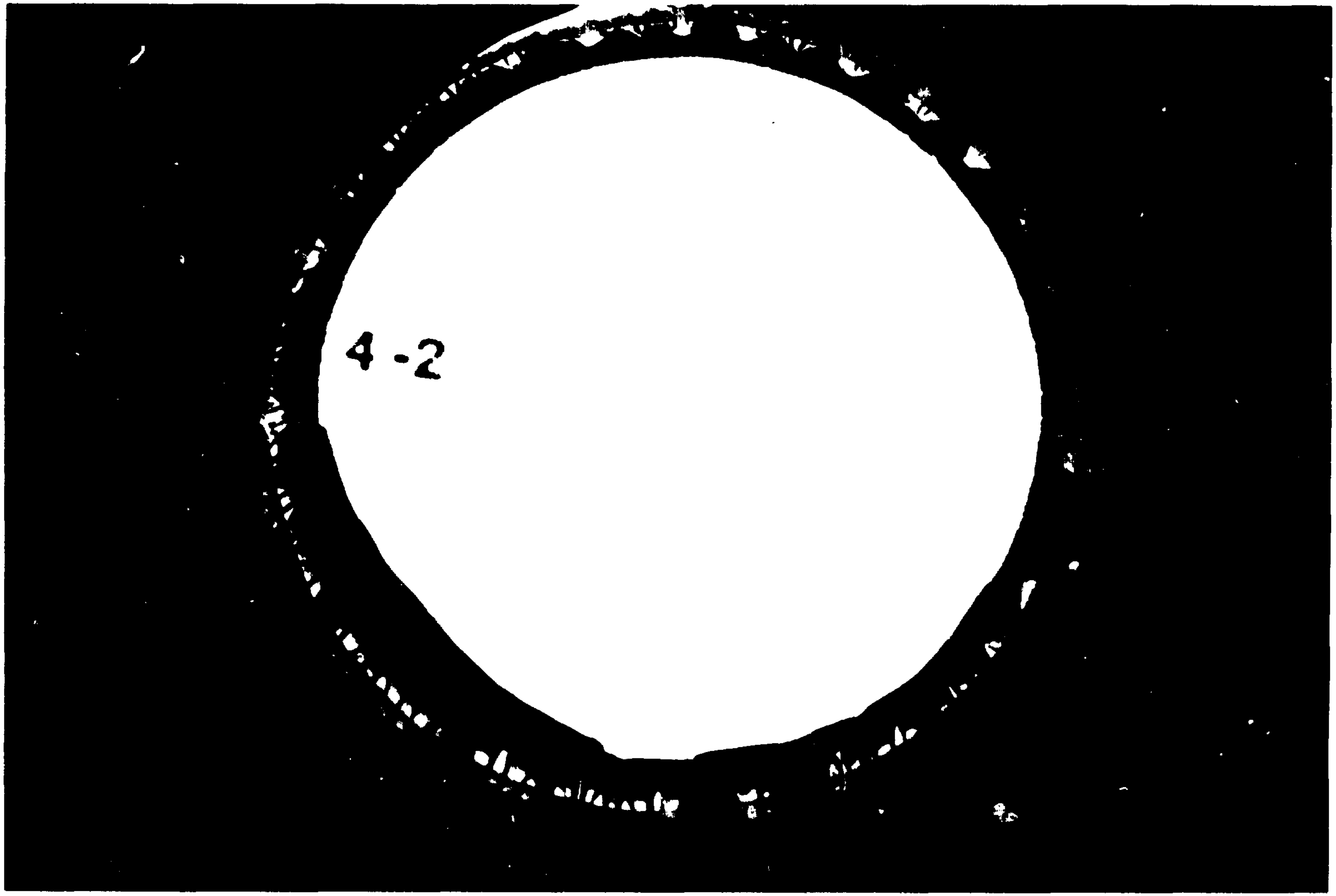

Fig. 18. Combination 4-2 neat-resin sample after cryogenic shock/soak

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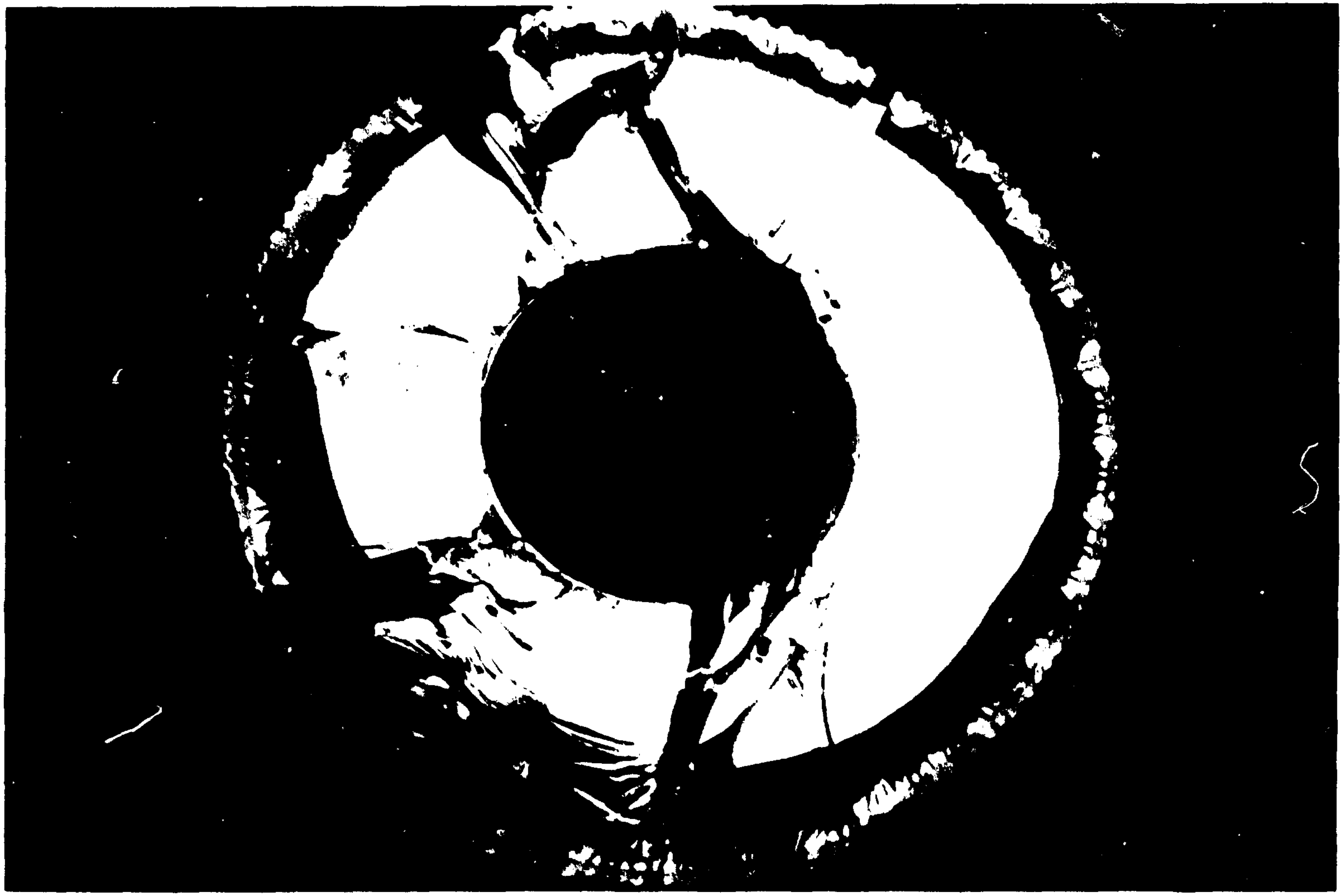

Fig. 19. Combination 4-2 embedment sample after cryogenic shock/soak

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(U)

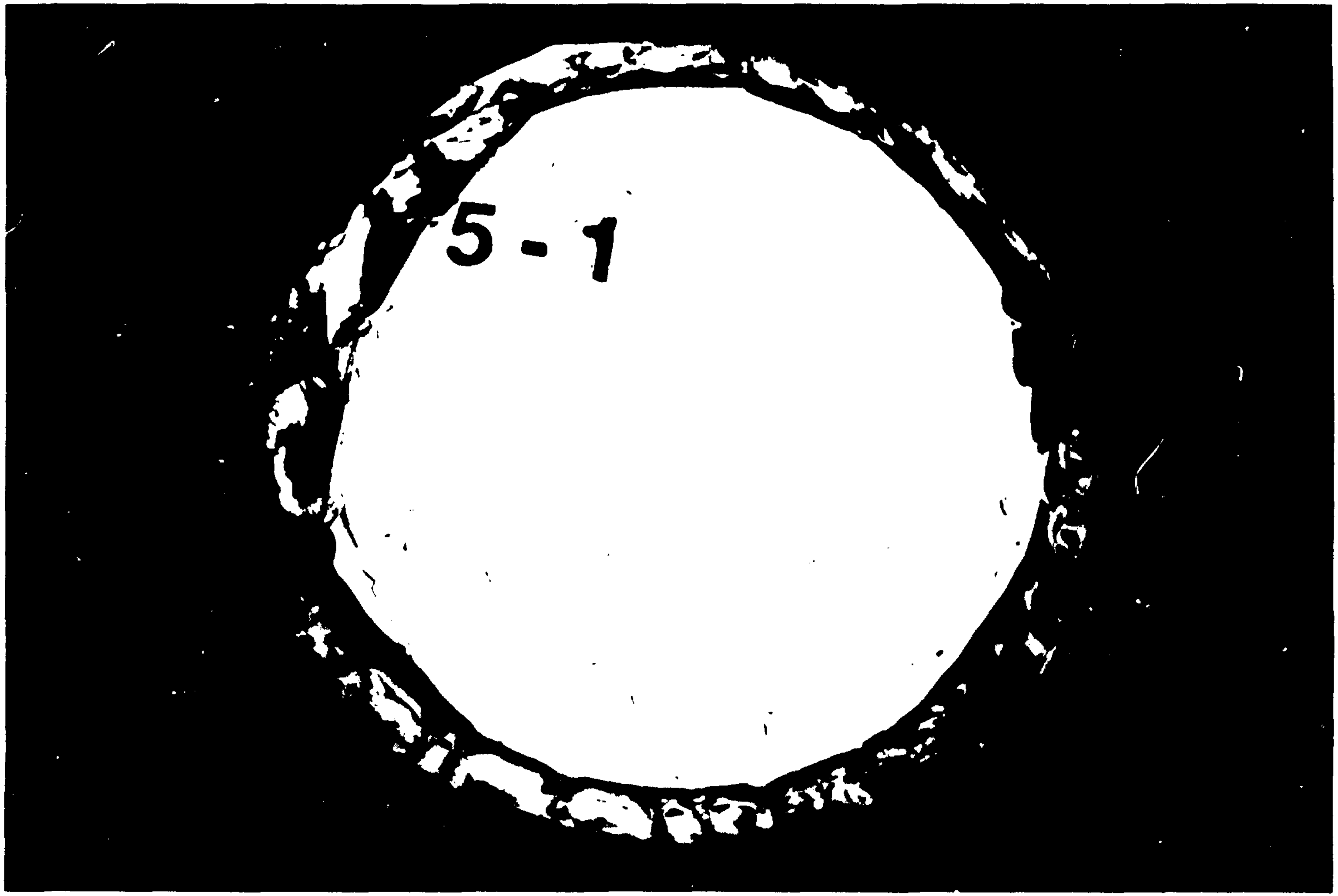

Fig. 20. Combination 5-1 neat-resin sample after cryogenic shock/soak

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(U)

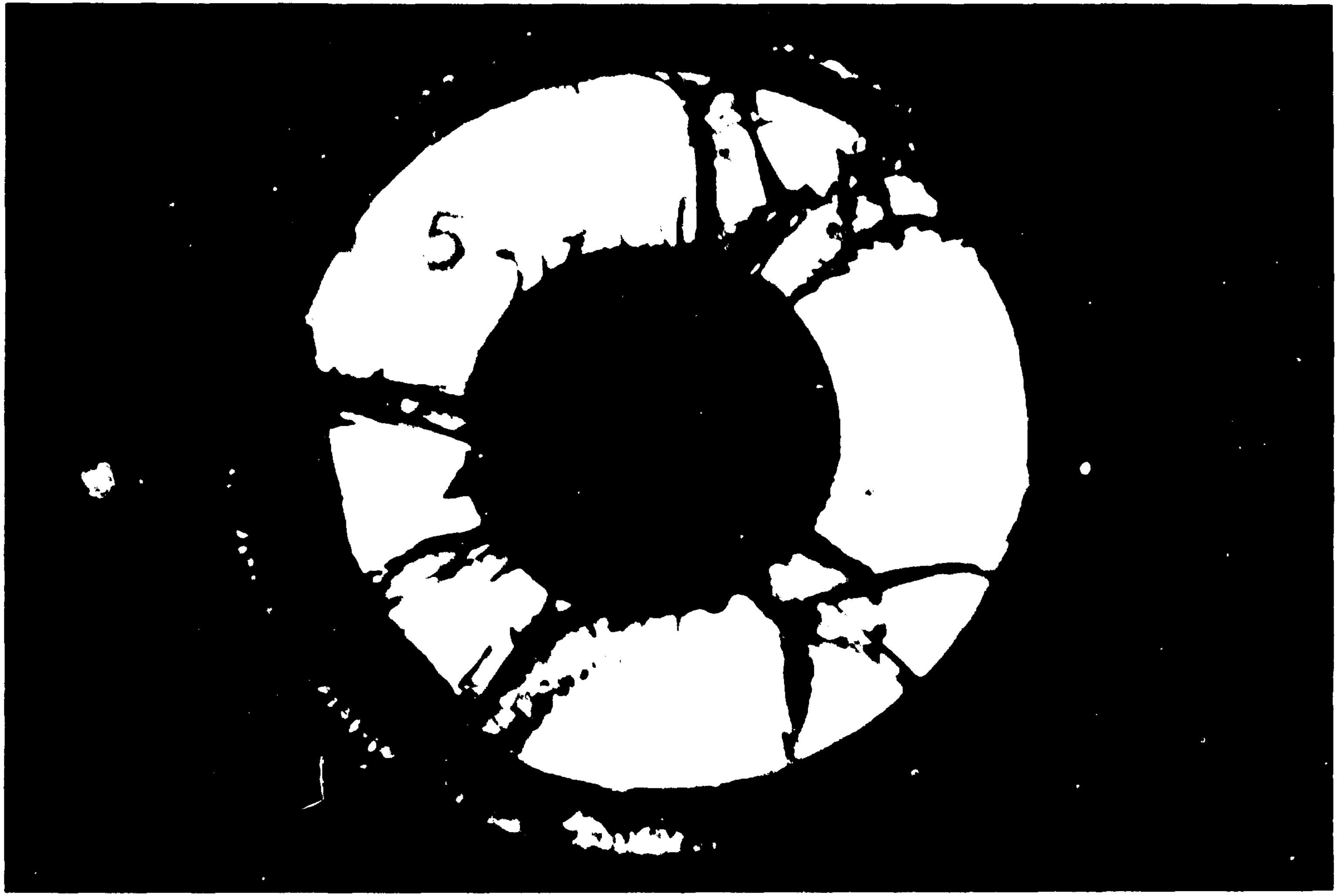

Fig. 21. Combination 5-1 embedment sample after cryogenic shock/soak

PHOTO NO. K/PH-90-0162
(U)

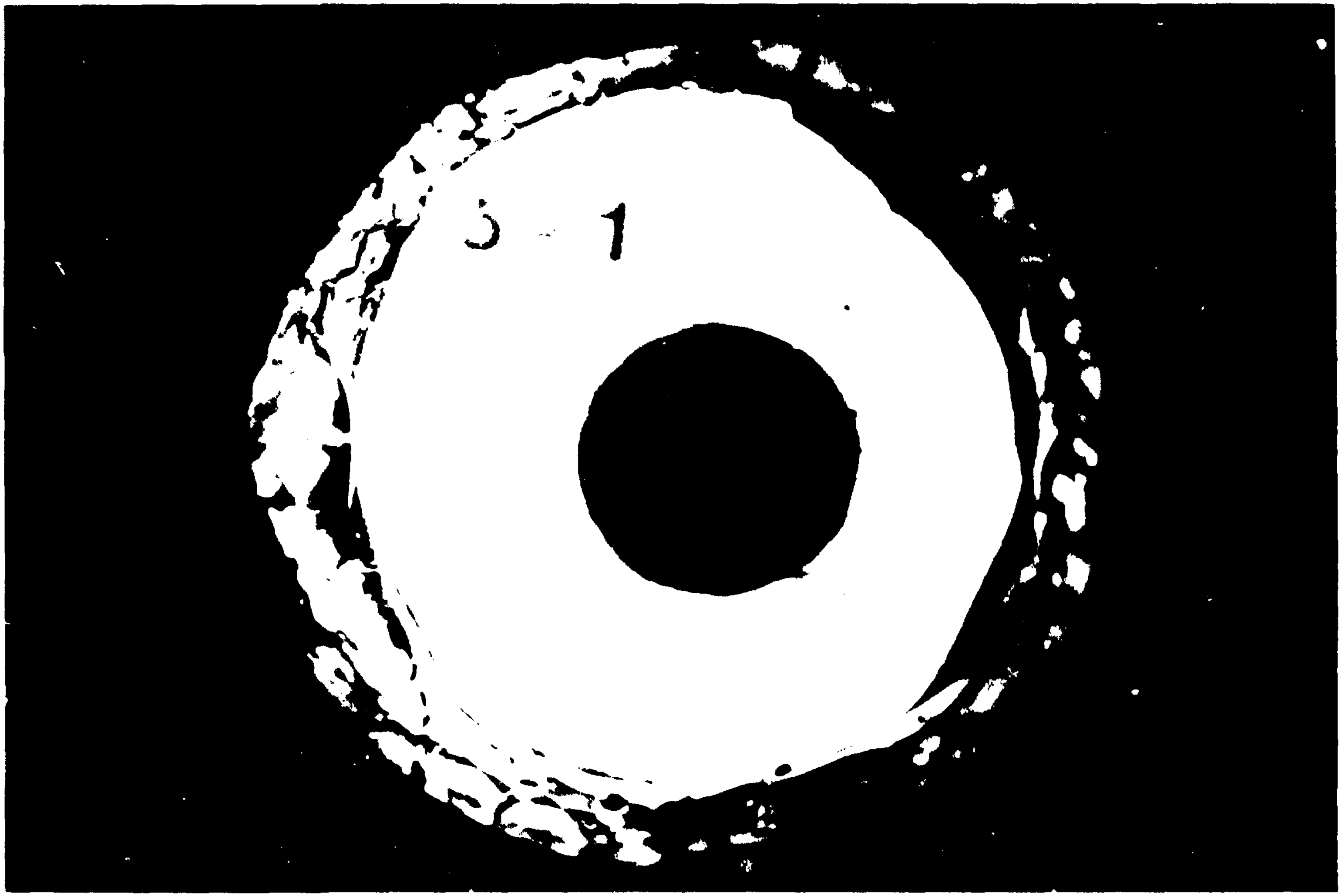

Fig. 22. Combination 5-1 reduced-volume embedment after cryogenic shock/soak

PHOTO NO. K/PH-80-0166
(U)

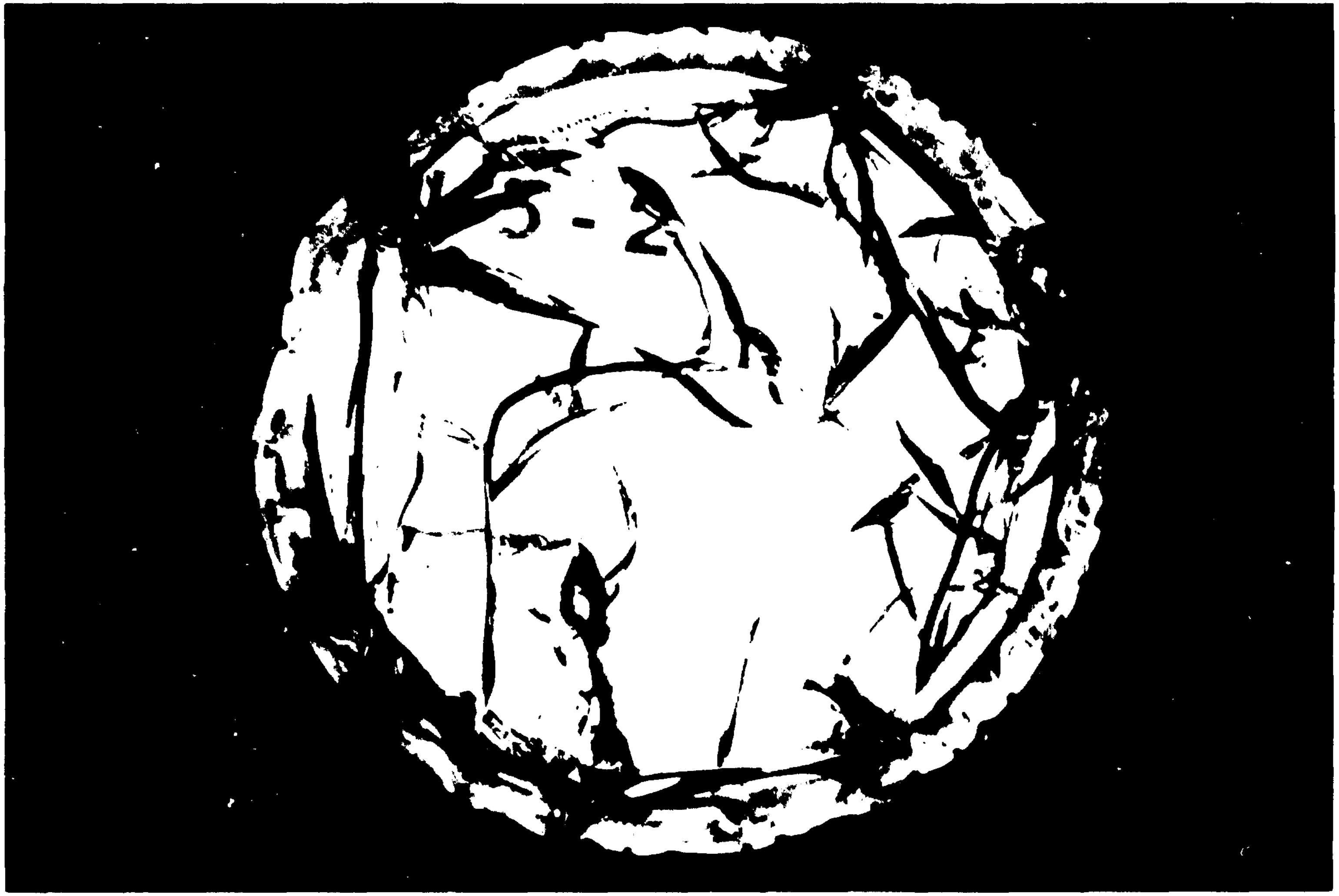

Fig. 23. Combination 5-2 neat-resin sample after cryogenic shock/soak

PHOTO NO. K/PH-90-0188
(U)

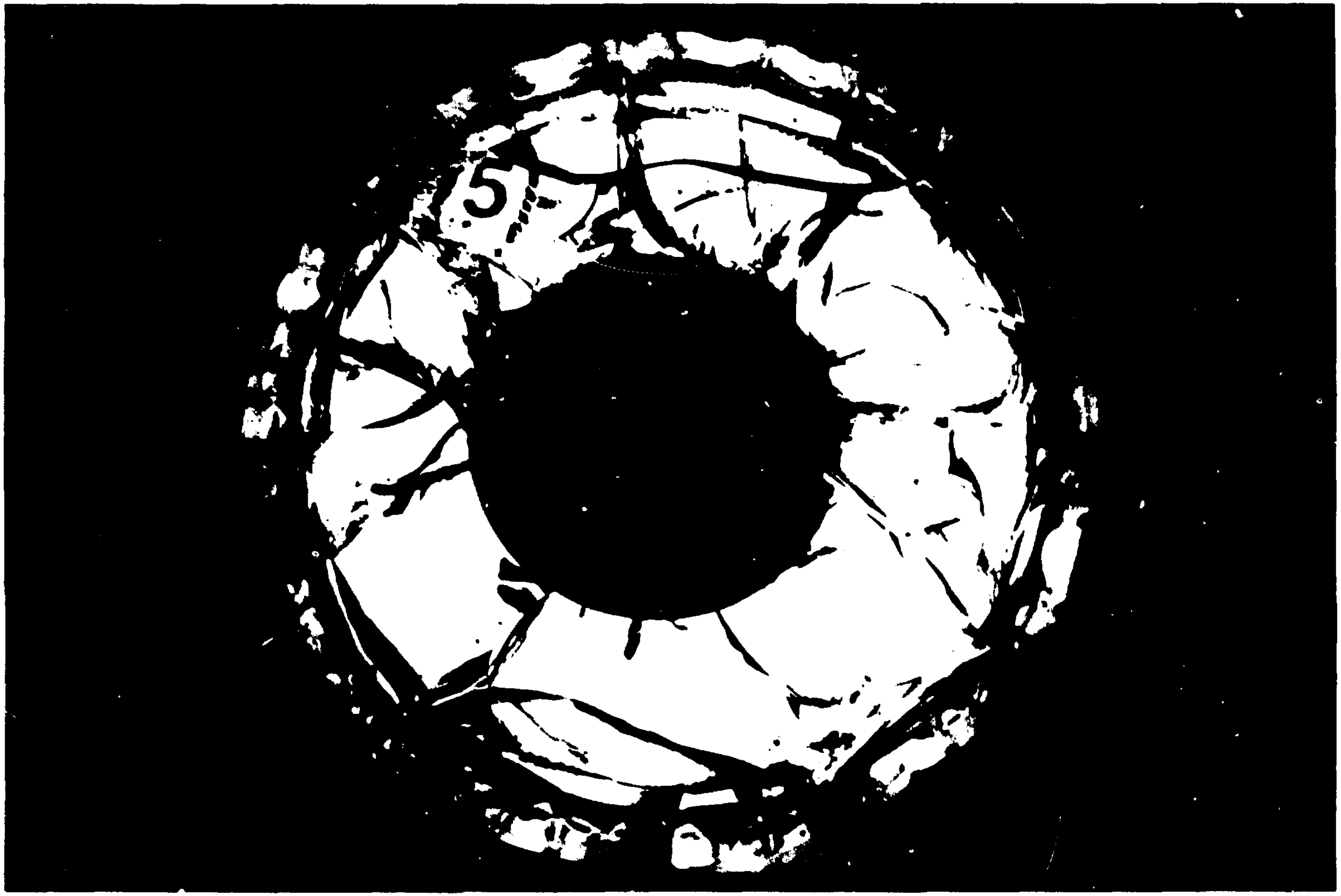

Fig. 24. Combination 5-2 embedment sample after cryogenic shock/soak

PHOTO NO. K/PH-80-0160
(U)

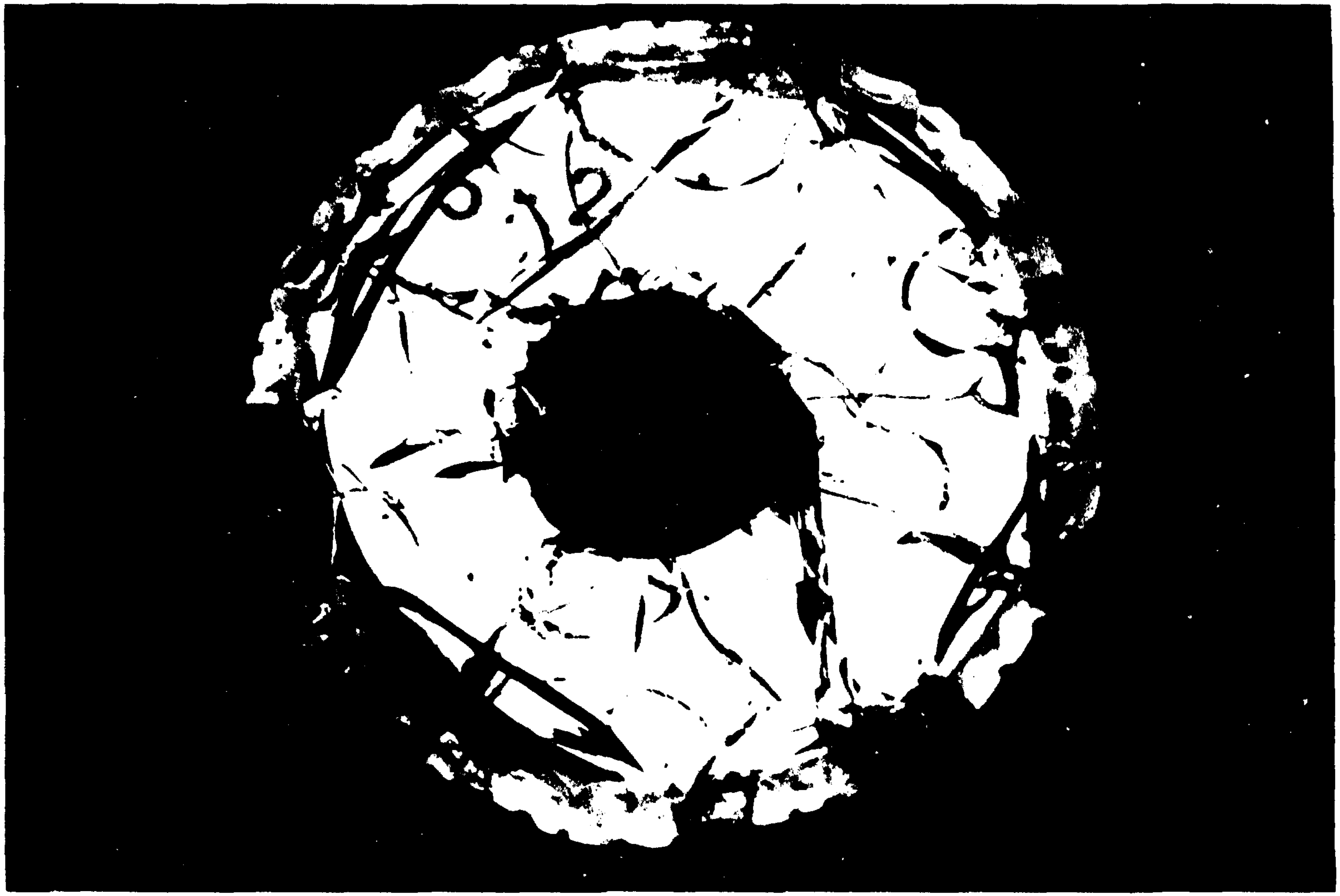

Fig. 25. Combination 5-2 reduced-volume embedment after cryogenic shock/soak

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(U)

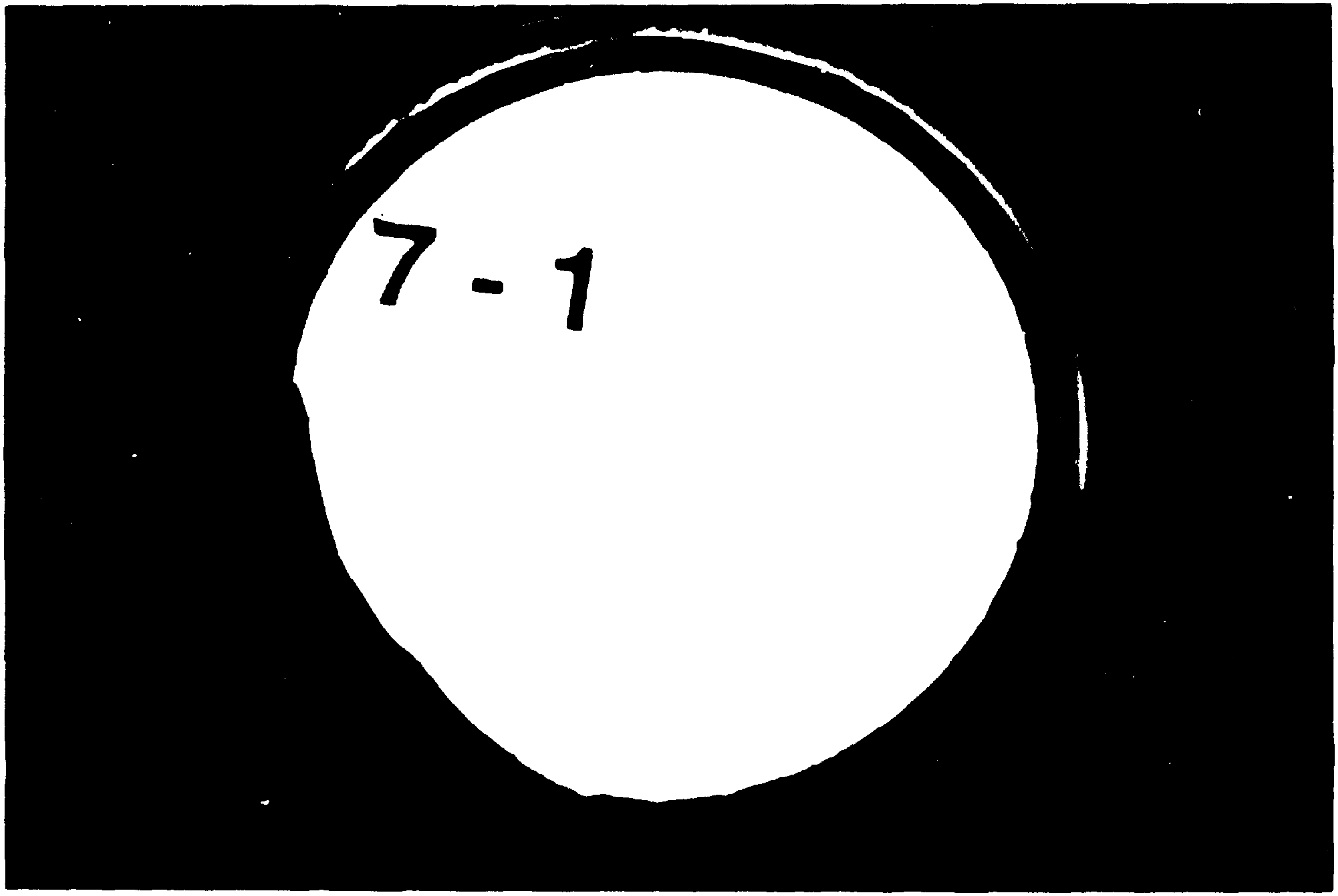

Fig. 26. Combination 7-1 neat-resin sample after cryogenic shock/soak

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(U)

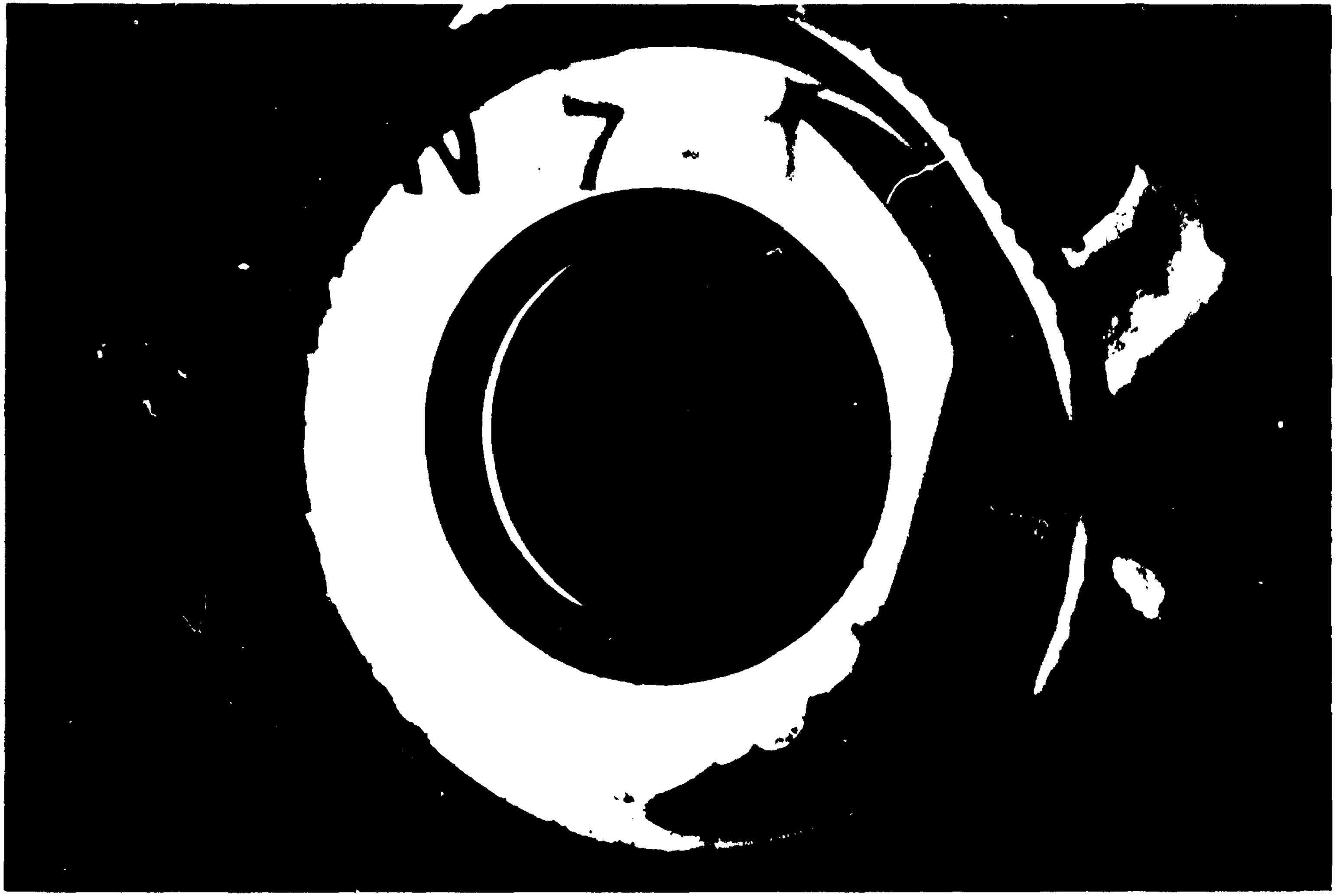

Fig. 27. Combination 7-1 embedment sample after cryogenic shock/soak; sample has been tilted to "shear crown" and embedment

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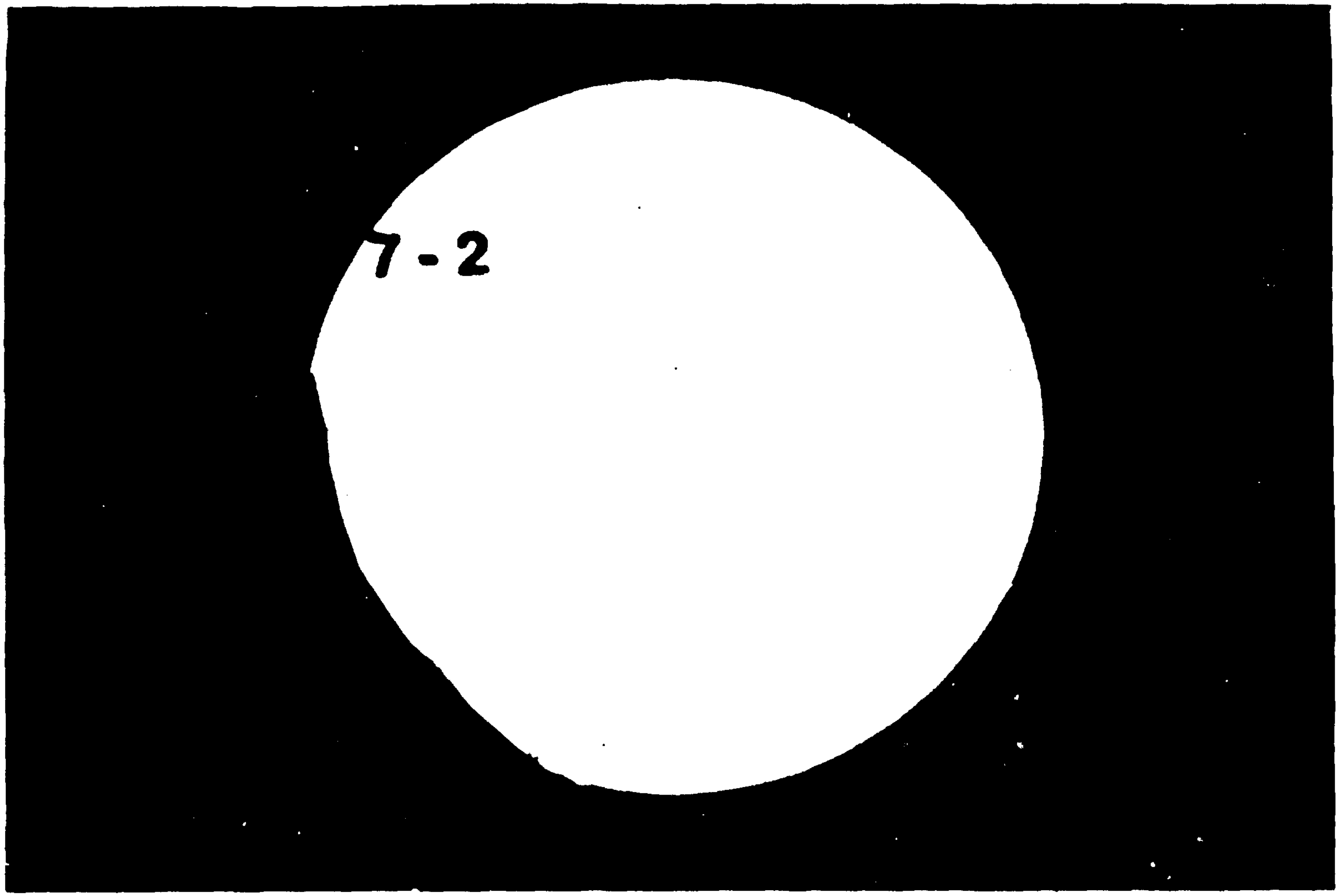

Fig. 28. Combination 7-2 neat-resin sample after cryogenic shock/soak

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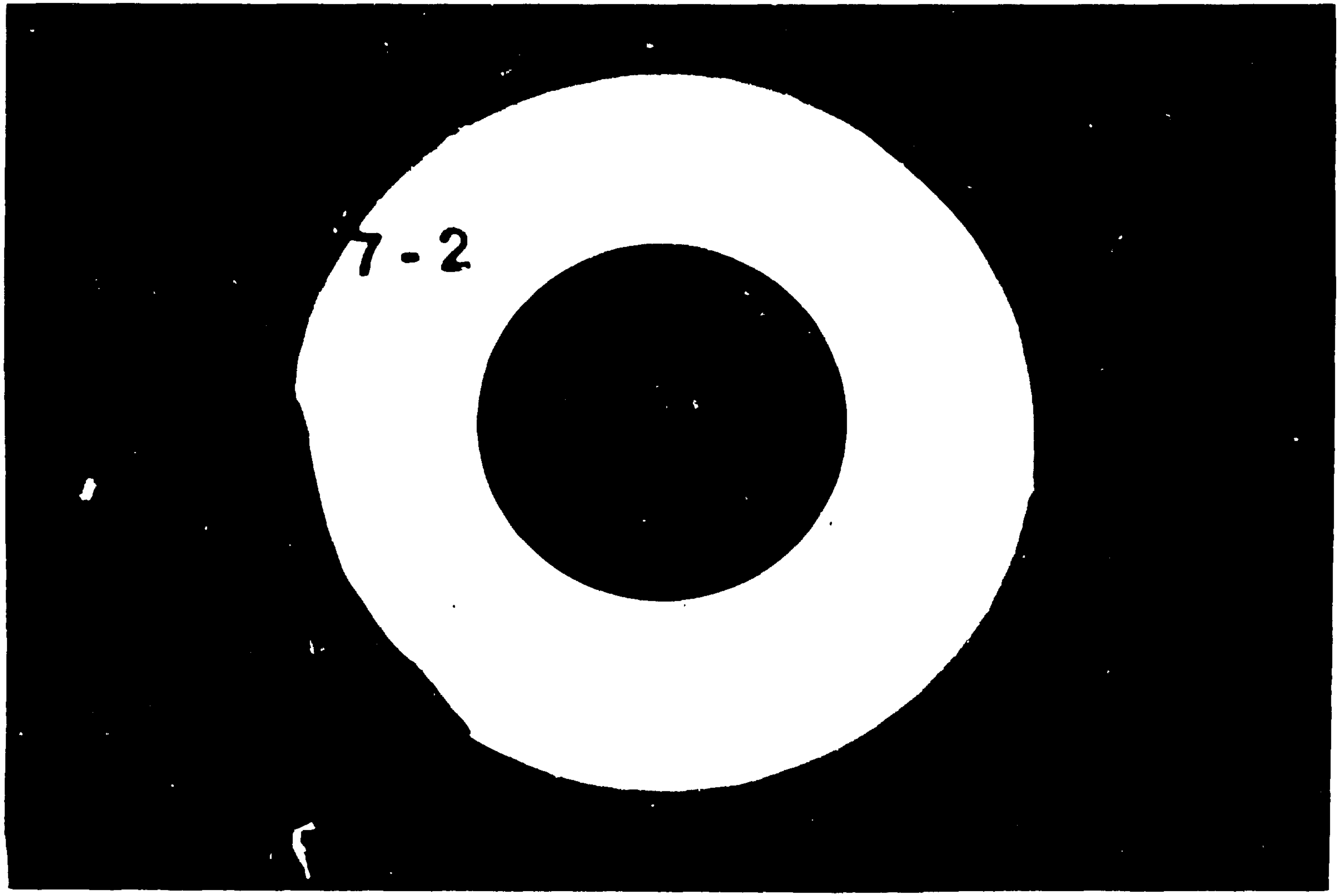

Fig. 29. Combination 7-2 embedment sample after cryogenic shock/soak

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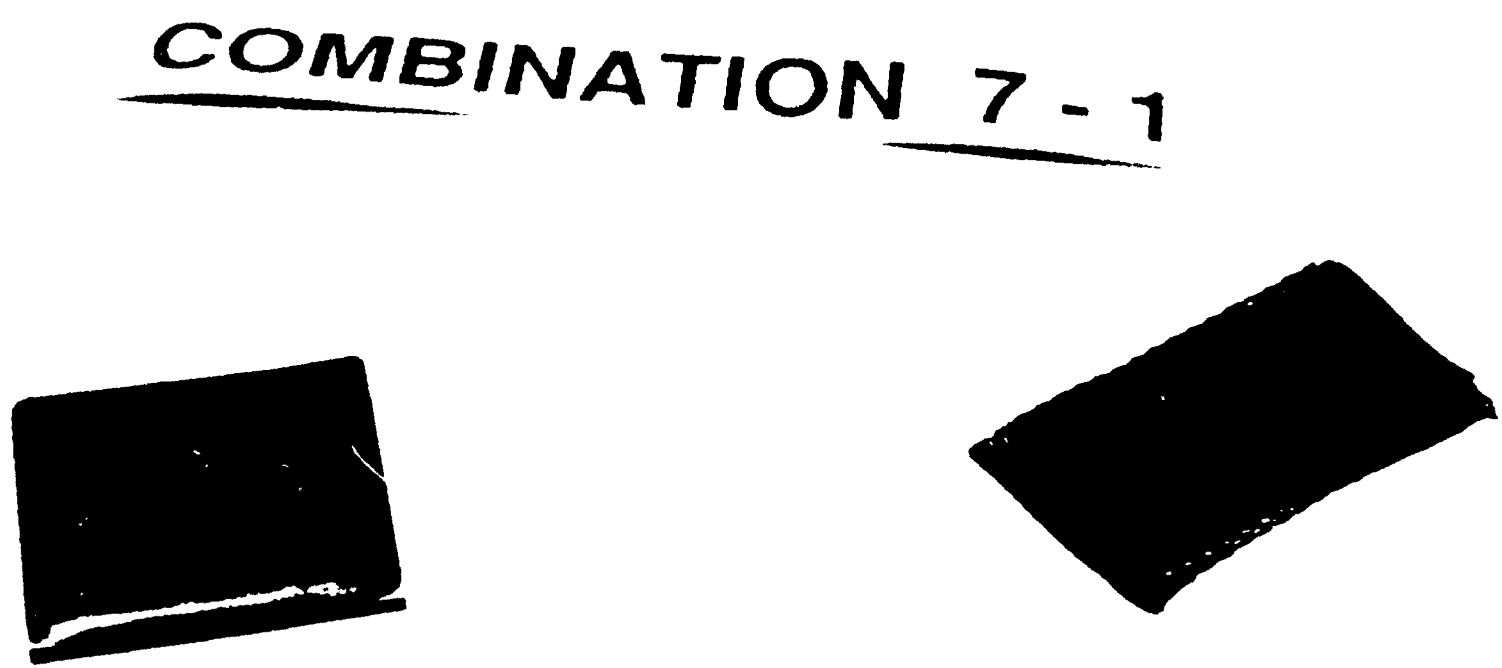

Fig. 30. Combination 7-1 wet-out samples. Note that drop outline is still evident on glass fabric, and Spaulrad™ sheet edge has not been reached by flow.

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(U)

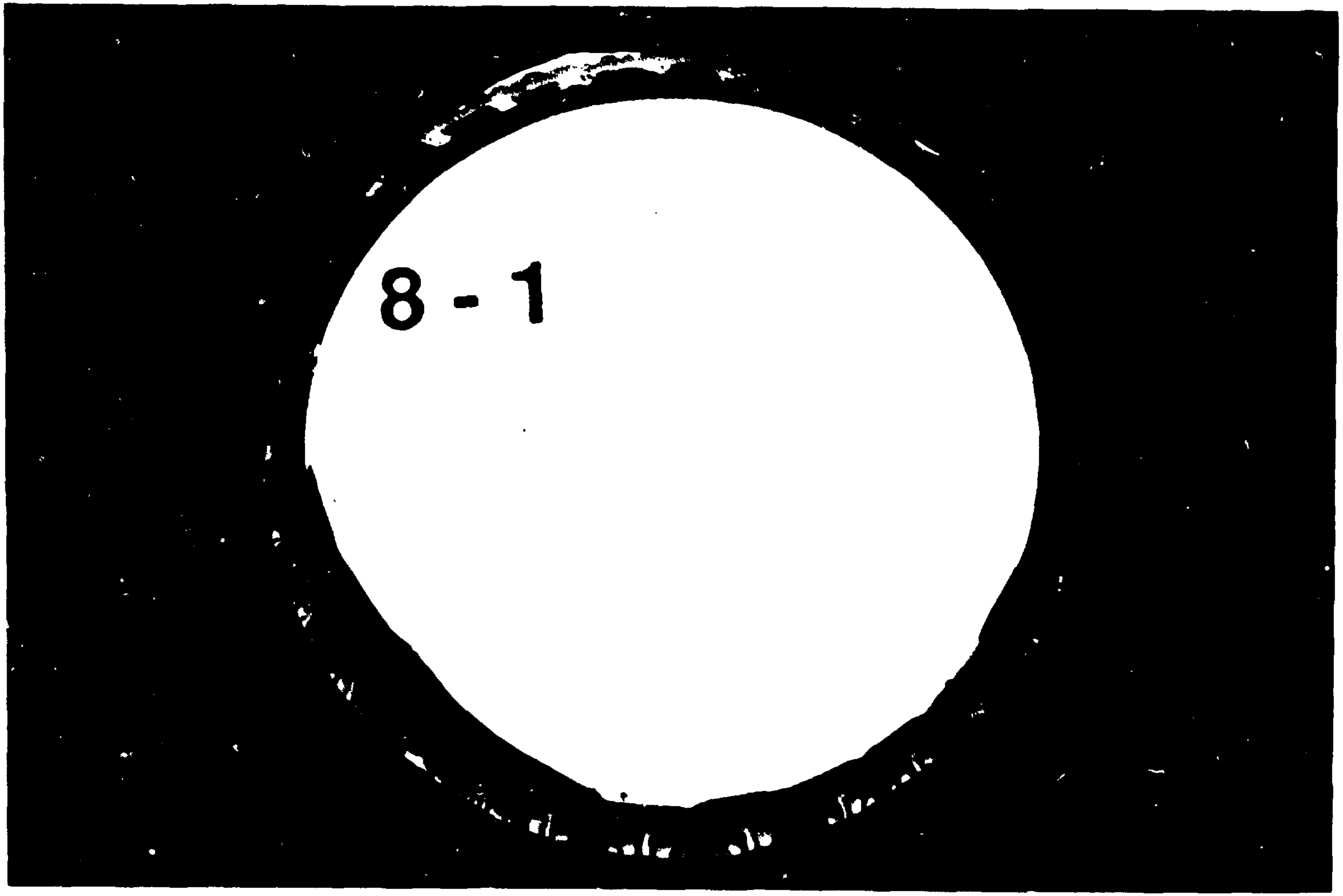

Fig. 31. Combination 8-1 neat-resin sample after cryogenic shock/soak

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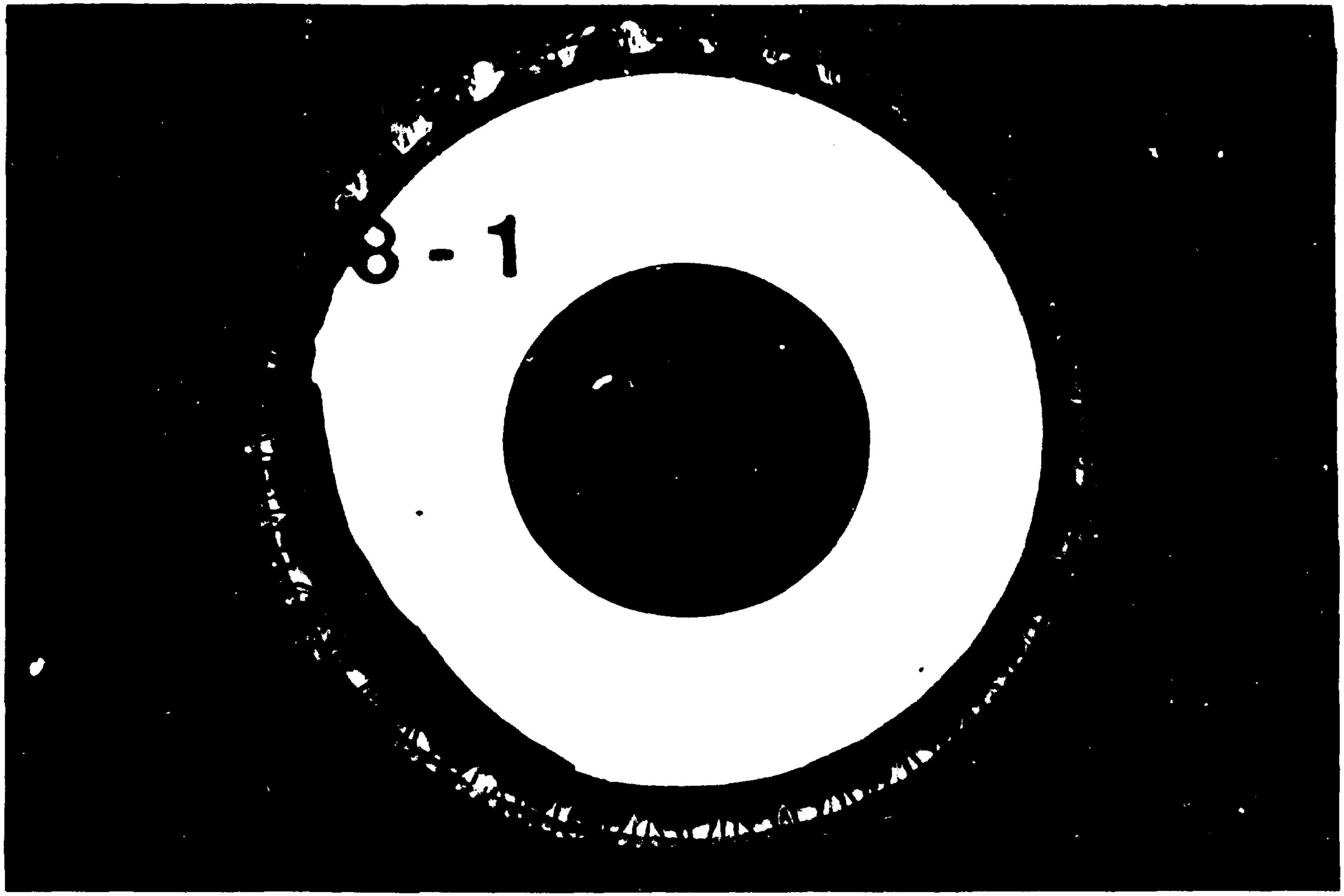

Fig. 32. Combination 8-1 embedment sample after cryogenic shock/soak

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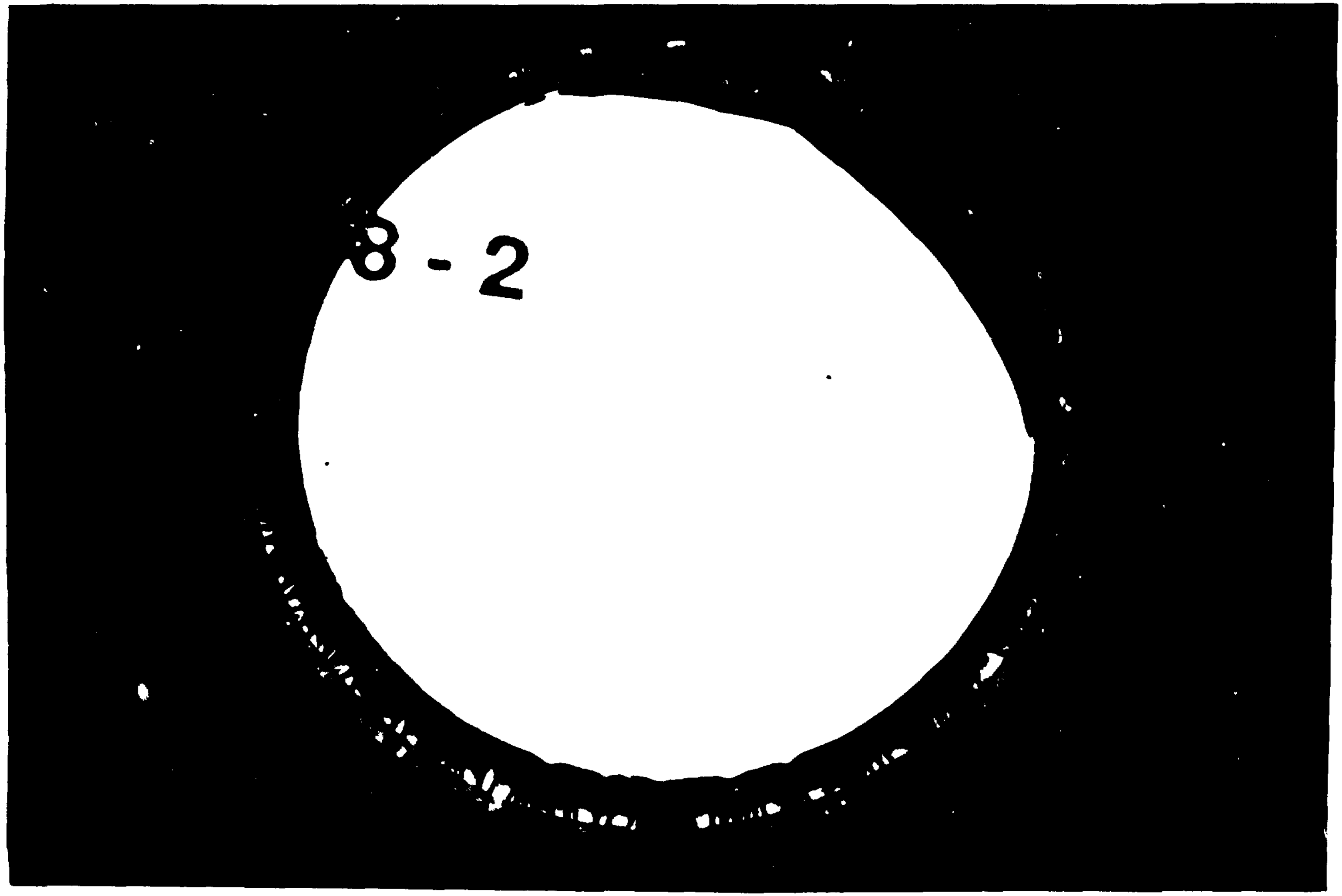

Fig. 33. Combination 8-2 neat-resin sample after cryogenic shock/soak

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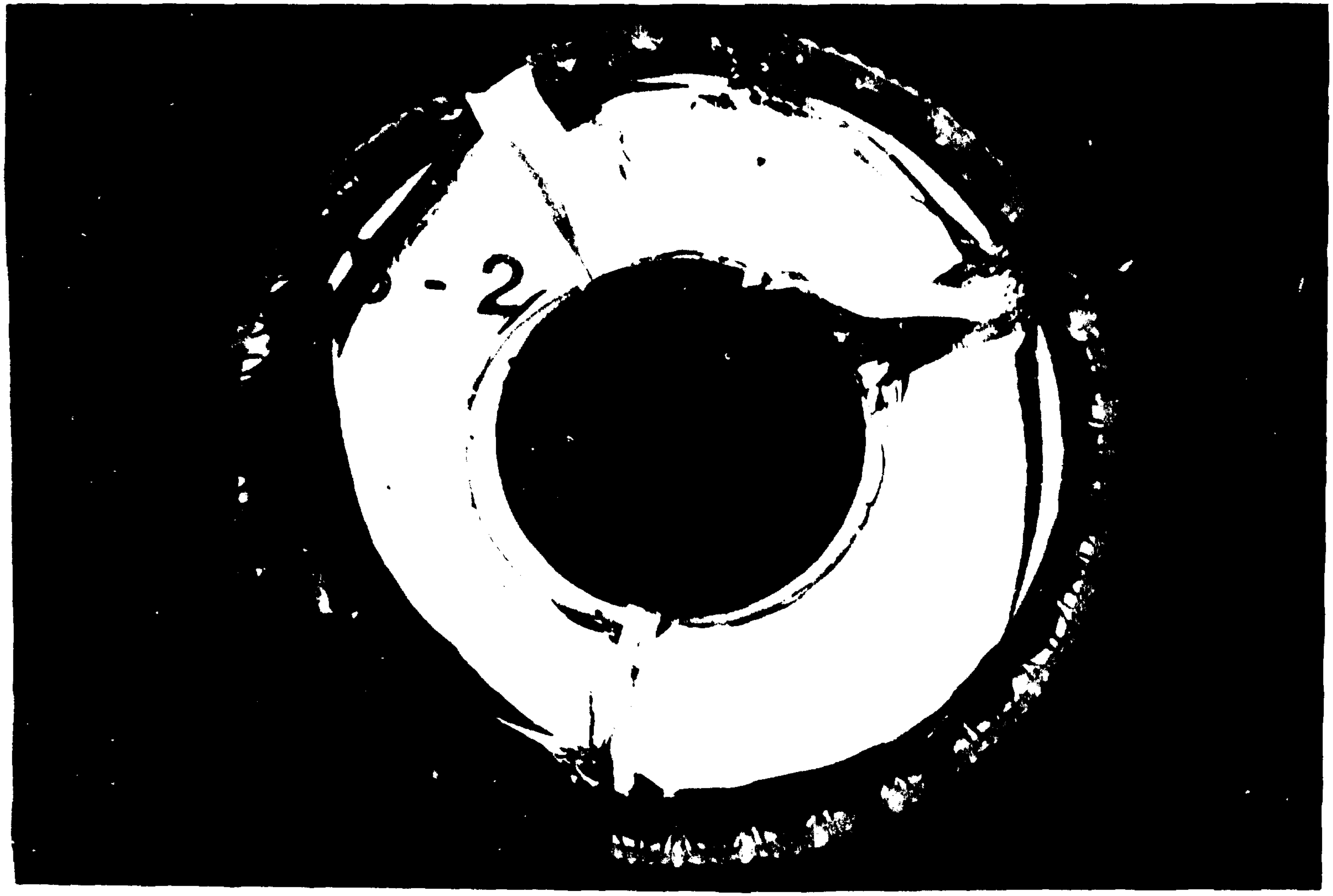

Fig. 34. Combination 8-2 embedment sample after cryogenic shock/soak