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MASTER

DESTRUCTIVE ANALYSIS OF NEUTRON SOURCES M-71 AND 1053S

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DESTRUCTIVE ANALYSIS OF NEUTRON SOURCES M-71 AND 1053S

I. Description and History of Sources

Destructive post mortem analyses were performed on two neutron sources #M-71 and #1053S. Figure #1 shows the inner and outer construction of this type of source. The outer container consisted of a 0.030 in. 304 stainless steel jacket with a 0.250 in. TIG welded SS lid. The inner container was a 0.070 in. tantalum jacket with a TIG welded 0.250 in. tantalum plug. The fuel form that was used was the intermetallic compound Pu Be_{13}.

Source M-71 was fabricated in October of 1957 and shipped to the University of California. It was subjected to a weapons test and in July of 1963, returned to Mound Laboratory for storage. Source #1053S was fabricated in 1962 and due to a defective inner liner was retained at Mound in storage.

A summary of the histories of the ten neutron sources that will be examined during this study is given in Appendix A. These two sources were chosen for examination first because they are among the worst cases. A summary of the status of work performed to date is given in Appendix B.

II. Test Procedure

Radiographic analysis was obtained on the sources prior to the metallographic examination. Tables I and II show the results of this analysis. A possible defect of the inner weld was seen in M-71. Evidence of the inner liner of 1053S as being completely destroyed was observed.

III. Sectioning Procedure

Sectioning of the two sources was carried out in a radioactive dry box that contained a once through nitrogen atmosphere. A circular saw with an aluminum oxide blade was utilized to make peripheral cut around the outer and inner containers of both sources. All welds, including representative sections of the top, sidewall and bottom of the outer and inner liner were taken for metallographic examination.

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IV. Metallographic Procedure

The samples were vacuum mounted with epoxy and were ground on 180, 320, and 600 grit silicon carbide papers. Polishing was accomplished on a Syntron vibratory polisher using a water slurry of 0.3 μm alumina on texmet polishing paper for 15 hours. Photomicrographs of etched and non-etched samples were taken on an MM-5 Leitz metallograph. The 304 stainless steel was etched electrolytically using a saturated solution of oxalic acid. The tantalum liner was not etched as investigation of the liner is incomplete at this time.

V. Results

Radiographic analysis of source M-71 as shown in Table I, indicated no defects were observed in the stainless steel outer container; however, the question did arise concerning the inner tantalum liner weld as being open. Table II shows no defects were observed in the stainless outer container of source 1053S; however, complete destruction of the inner tantalum liner side wall was readily apparent.

Various observations were made during the cross sectioning of the two sources. The inside of the stainless outer containers of both capsules appeared very clean, when removed from around the inner liner. No evidence of any reaction product or oxide layer was observed. The inner container of source M-71 also appeared very clean with no apparent ruptured zones, although there did appear to be a hairline crack in the top weld. The inner liner of source 1053S was completely covered with a greenish colored oxide. One side of the side wall was completely ruptured. A peripheral cut was made on both inner liners. Source M-71 is shown in Figure 2. Note that the tantalum plug did break away, indicating a crack may have existed in the weld zone. Figures 3 and 4 show photographs of the ruptured areas and the extent of cracking of source 1053S. The fuel in both sources appeared very metallic silver in color. Large holes were observed in the fuel of both sources as seen in Figure 2 and 4 indicating the possibility of gas bubbles being formed while the fuel was molten during the fabrication process.

Photomicrographs of the unetched and etched structures of the outer (SS) container welds of M-71 and 1053S were taken and are shown in Figures 5-12. Cracking can be observed in the

weld zones of both sources. Figure 13 shows the cracking through the weld zone of the inner tantalum container of M-71. A reaction product on the outside wall of M-71 weld area is shown in Figure 14.

Representative microphotographs of the inner tantalum liners are shown in Figures 15-18. Intergranular attack, cracking, and complete penetration of the side wall is readily apparent. Figure 18 shows penetration of the side wall has occurred, although this was not indicated by radiography. Figure 19 is typical of the fuel showing the porosity that exists. Full investigation of the inner liners is incomplete at this time as x-ray microprobe analysis is required.

Microhardness data, as shown in Figure 20, was typical of the values obtained for the top edge and side walls of the outer stainless steel container. Figures 21 and 22 show photomicrographs of the areas tested. The differences in the hardness that exist may be attributed to the cold working and the small grain size of the outer stainless lid in Figure 21, as compared with the annealed side wall in Figure 22. Figure 23 represents the hardness of the stainless outer top and side wall of M-71. These larger differences are unaccountable at this time, but are probably related to the reasons already stated. This source was subjected to a severe overpressure which may have contributed to the greater hardness differences.

VI. Correlation with NDT

Some correlation does exist between the radiographic results in Tables I and II with the results obtained from destructive analysis. A good indication as to the integrity of the outer and inner containers was realized although some cracks were observed metallographically which were not seen by radiography.

VII. Conclusions

The results obtained from the destructive analysis of neutron sources M-71 and 1053S indicate gross reactions taking place between the $PuBe_{13}$ fuel form and the tantalum inner container. Evidence of high pressures was observed in the porosity of the fuel and the cracking of weld zones of both the inner and outer containers. A partial correlation of the radiographic data with the destructive analyses was realized. The use of NDT analyses could be important on future investigations of neutron sources.

Date: 4-16-71

TABLE I

RADIOGRAPHIC INSPECTION REPORT

Item: NEUTRON SOURCE (PUBE)

Two radiographic views were made at 90° intervals to determine the general condition of the item.

Inspection results are as follows:

S/N- M75

OUTER

(1) No indication of defects in the outer container.

(2) Minimum weld if the outer container approximately 0.025".

INNER

(1) Possibility of some fuel-liner corrosion.

(2) Minimum weld of liner approximately 0.025".

FUEL

(1) Most uniform distribution of fuel 3/8" from end of liner adjacent to the tapped hole of the outer container.

Inspected by: James A. Stockton

Approval: W. S. Gray

Date: 4-16-71

TABLE II

RADIOGRAPHIC INSPECTION REPORT

Item: NEUTRON SOURCE (PUBE)

Two radiographic views were made at 90° intervals to determine the general condition of the item.

Inspection results are as follows:

S/N- M71

OUTER

(1) No indication of defects in the outer container.

(2) Minimum weld of the outer container approximately 0.040".

INNER

(1) No apparent fuel-liner corrosion.

(2) Liner weld joint possibly open.

FUEL

(1) Most uniform distribution of fuel ½" from end of liner adjacent to the tapped hole of outer container.

Inspected by: James A. Stockton

Approval: H. S. Gray

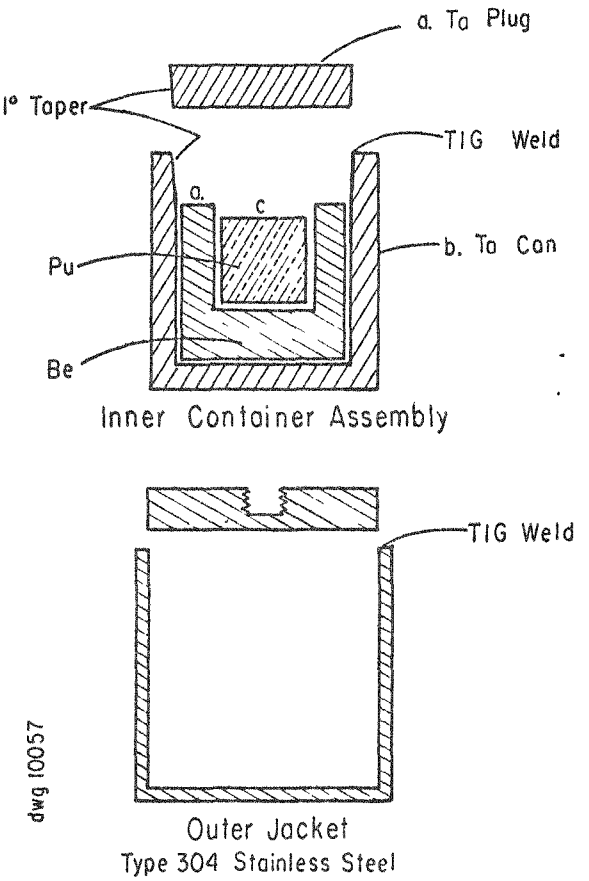

Figure 1 *PuBe Source Assembly*

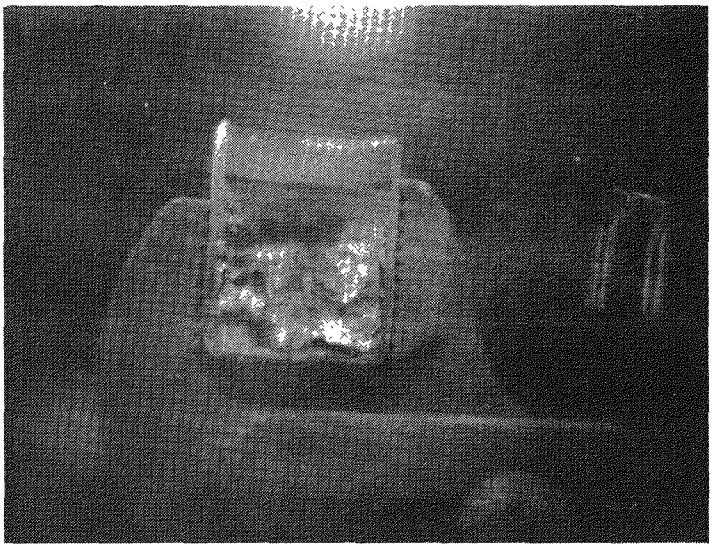

FIGURE 2. Photograph of Source M71 in Glovebox Approx. 1X

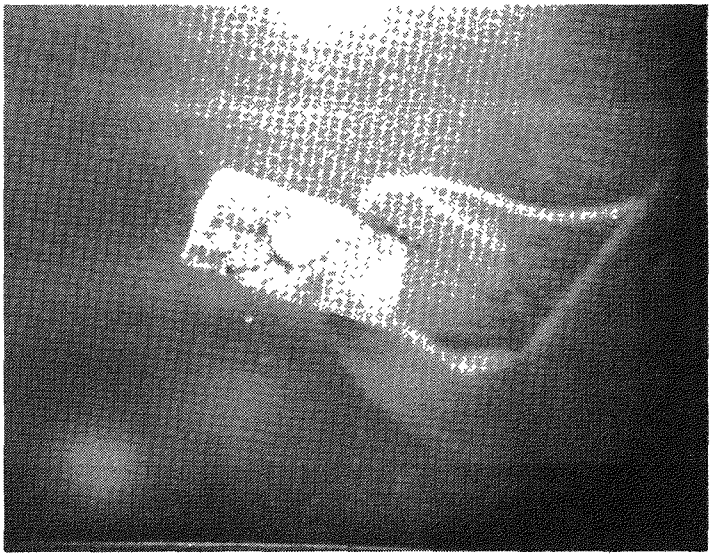

FIGURE 3. Photograph of Source 1053S in Glovebox Approx. 1X

FIGURE 4. Photograph of Source 1053S in Glovebox Approx. 1X

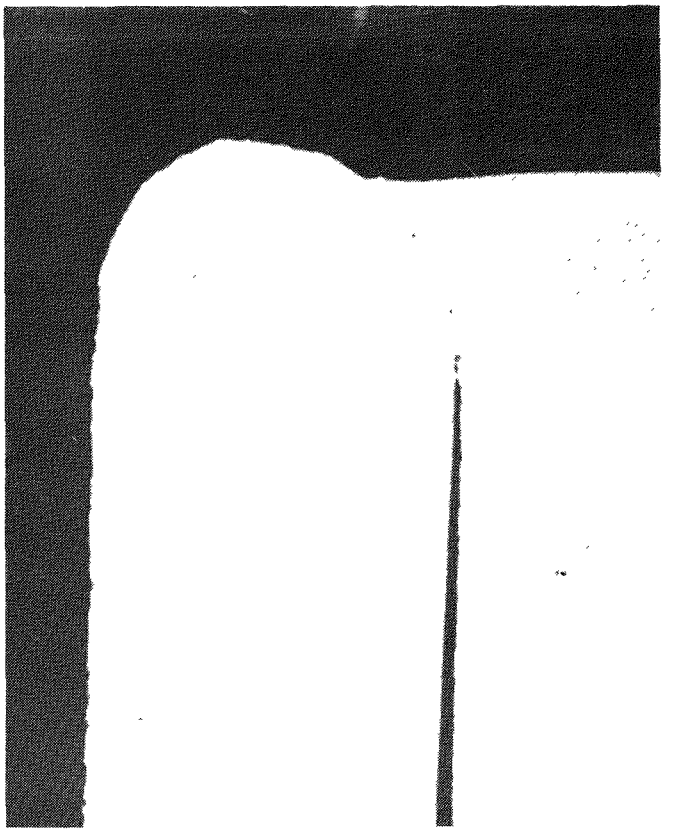

FIGURE 5. Photomicrographs of Unetched and Etched Weld Zones of M71
Top Weld - SS Outer Container
Source M71 60.5X

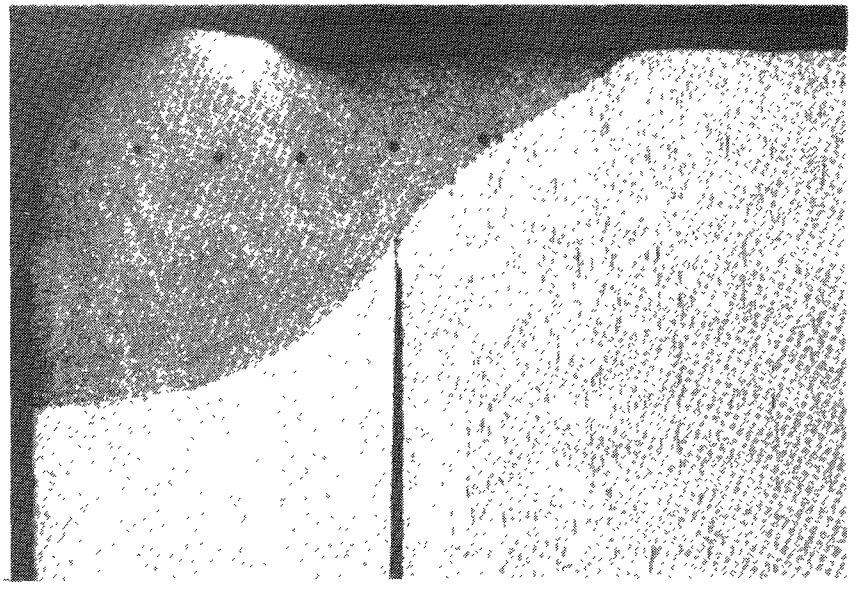

FIGURE 6. Photomicrographs of Unetched and Etched Weld Zones of M71
Top Weld - SS Outer Container
Source M71 60.5X

FIGURE 7. Photomicrographs of Unetched Weld Zones of M71 Bottom Weld - SS Outer Container 60.5X

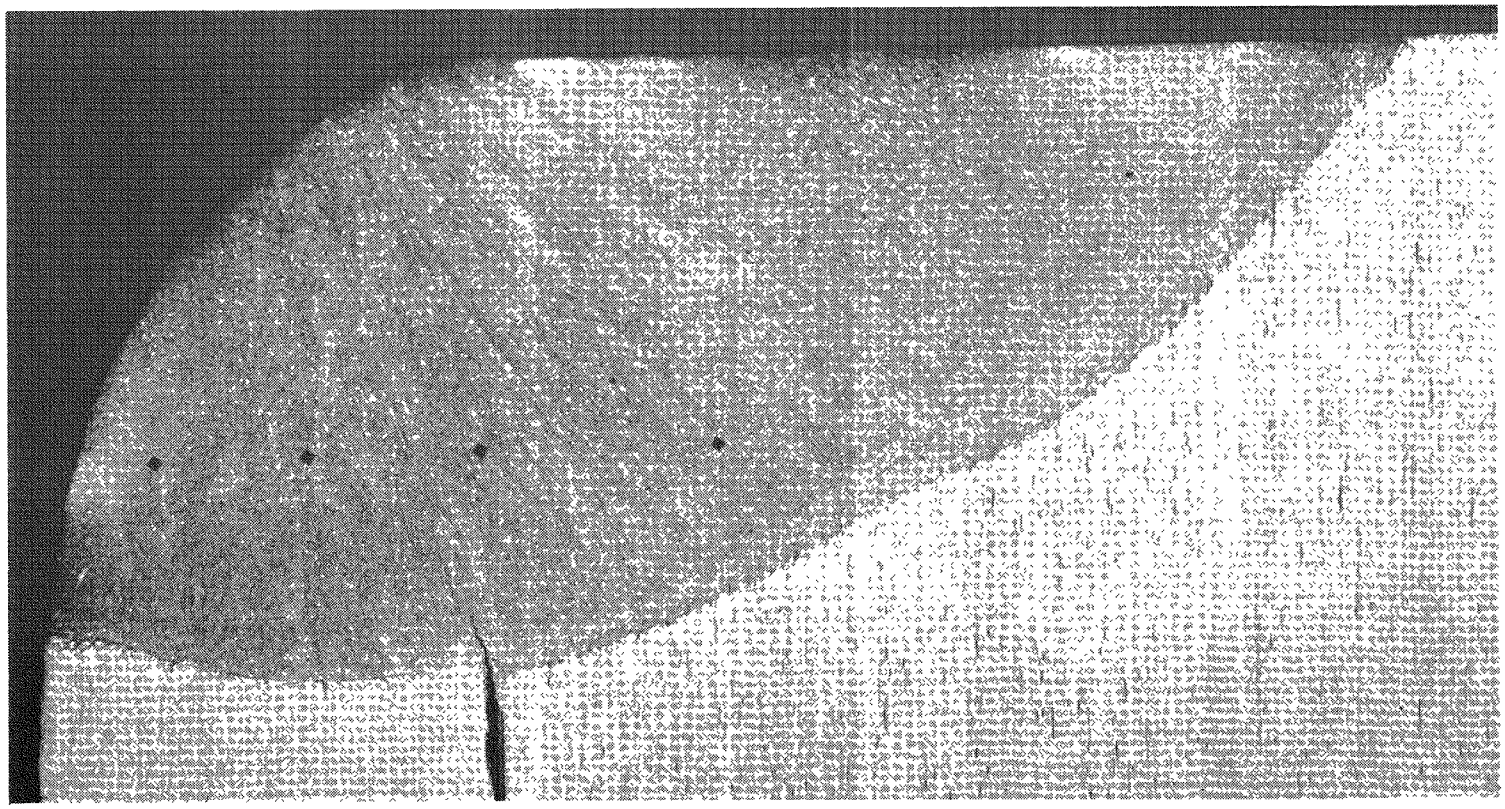

FIGURE 8. Photomicrographs of Etched Weld Zones of M71
Bottom Weld - SS Outer Container
60.5X

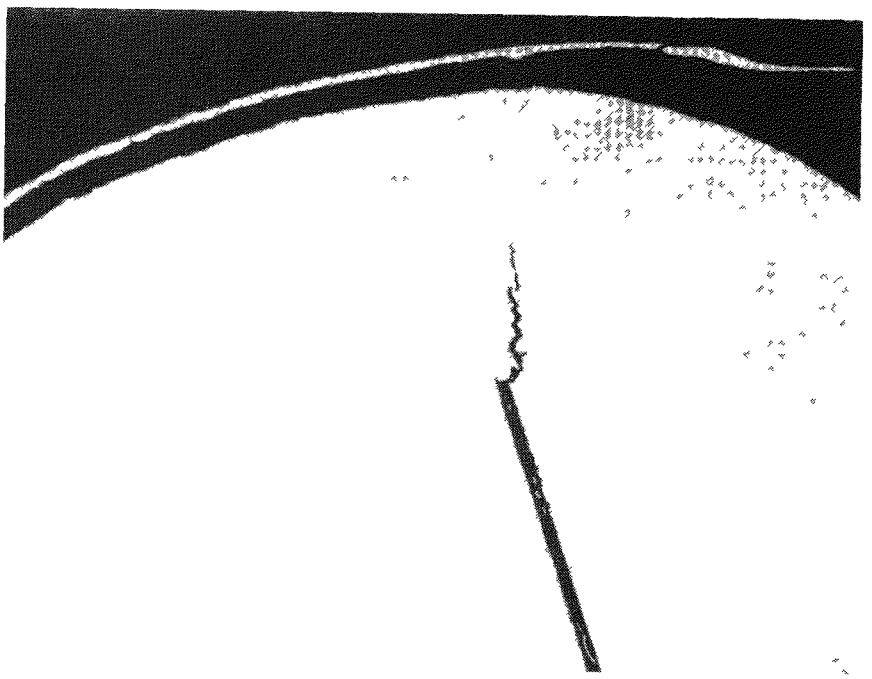

FIGURE 9. Photomicrographs of Unetched Weld Zones of 1053S
Top Weld - SS Outer Container
60.5X

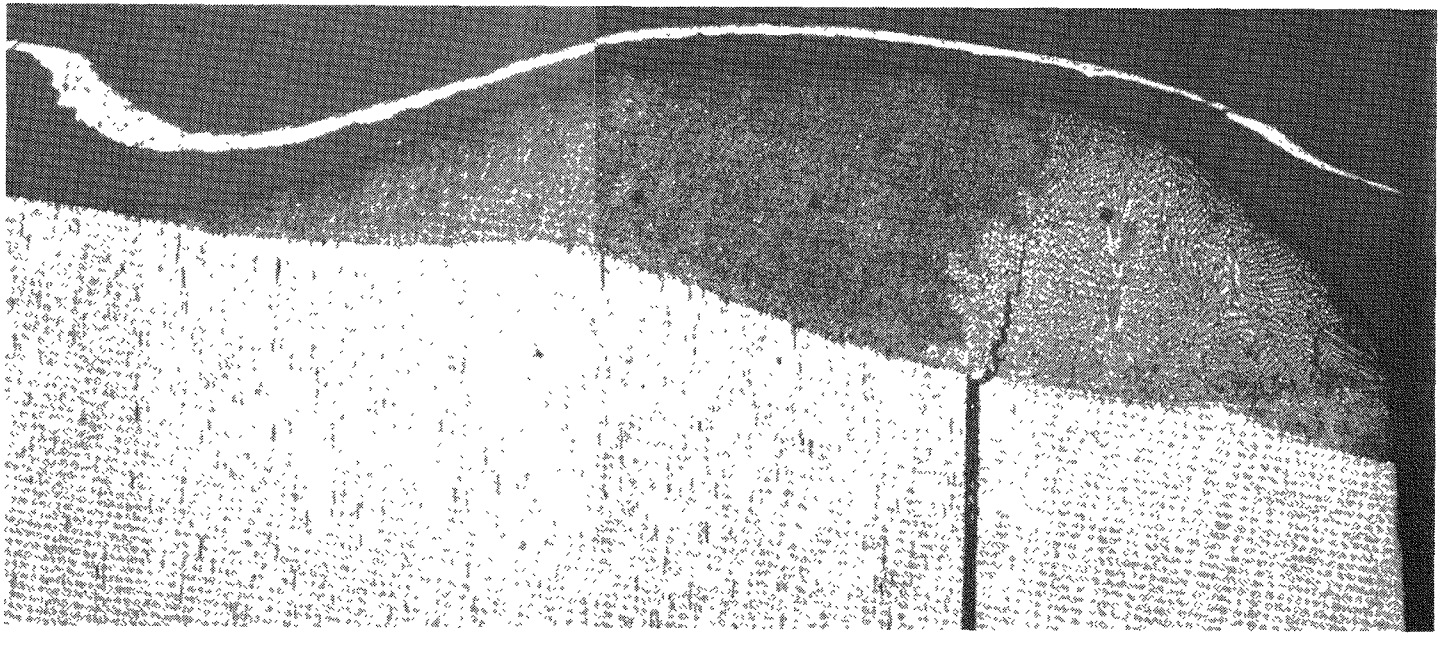

FIGURE 10. Photomicrograph of Etched Weld Zones of 1053S
Top Weld - SS Outer Container
60.5X

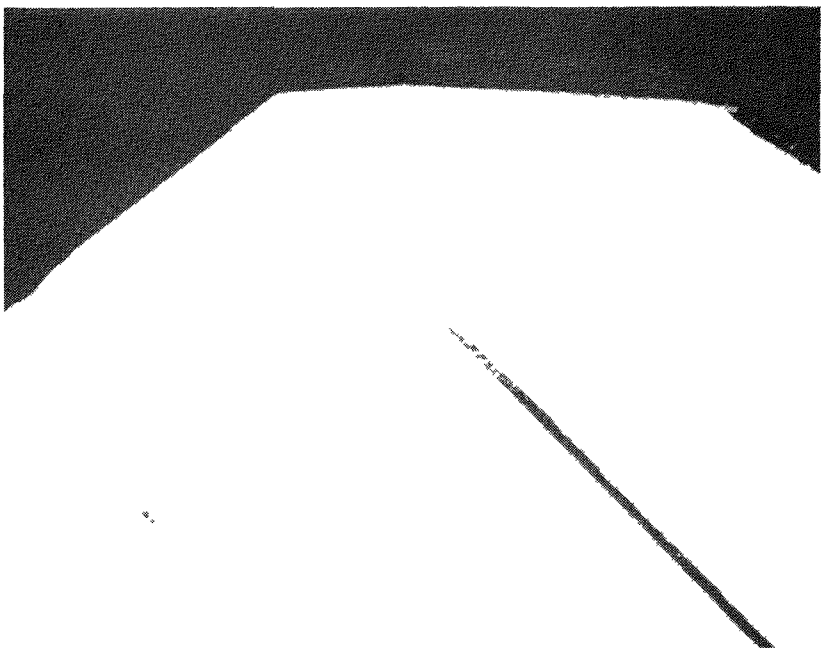

FIGURE 11. Photomicrograph of Unetched Weld Zones of 1053S
Bottom Weld - SS Outer Container
60.5X

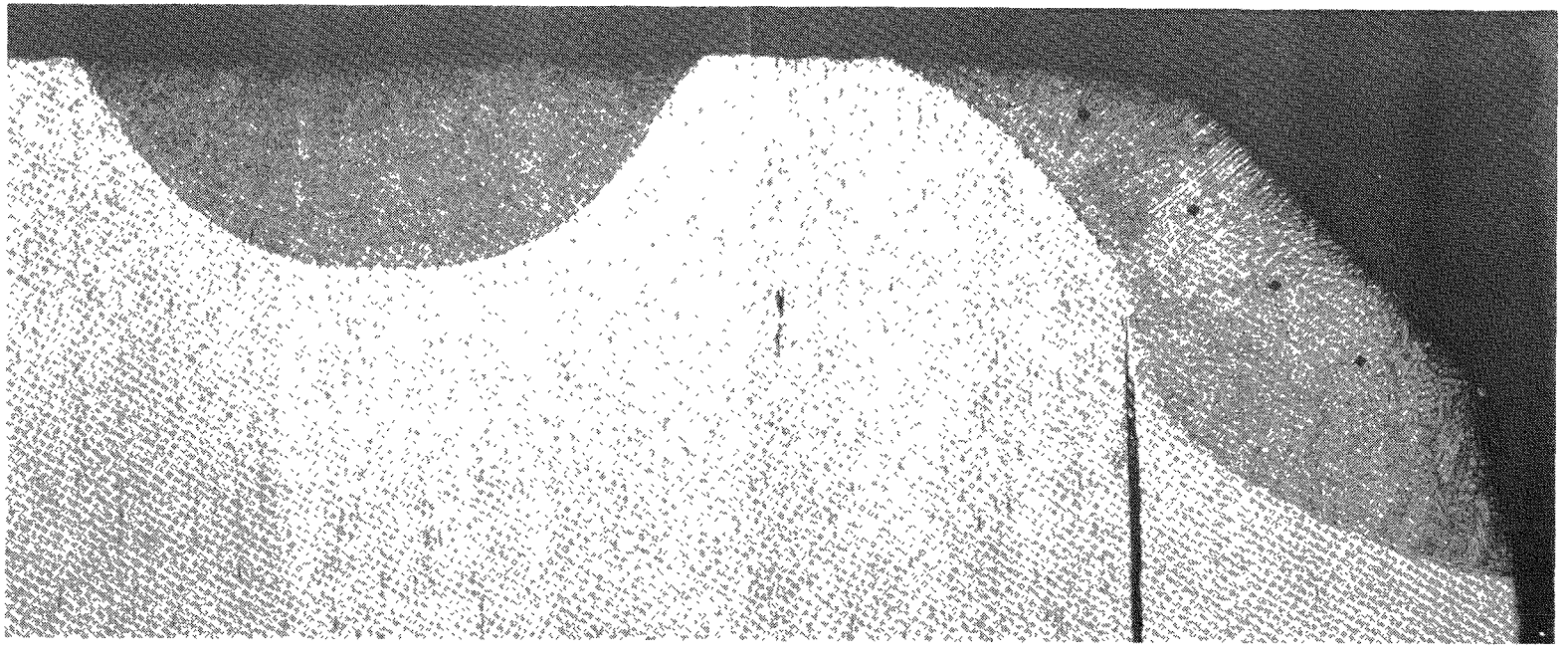

FIGURE 12. Photomicrograph of Etched Weld Zones of 1053S Bottom Weld - SS Outer Container
60.5X

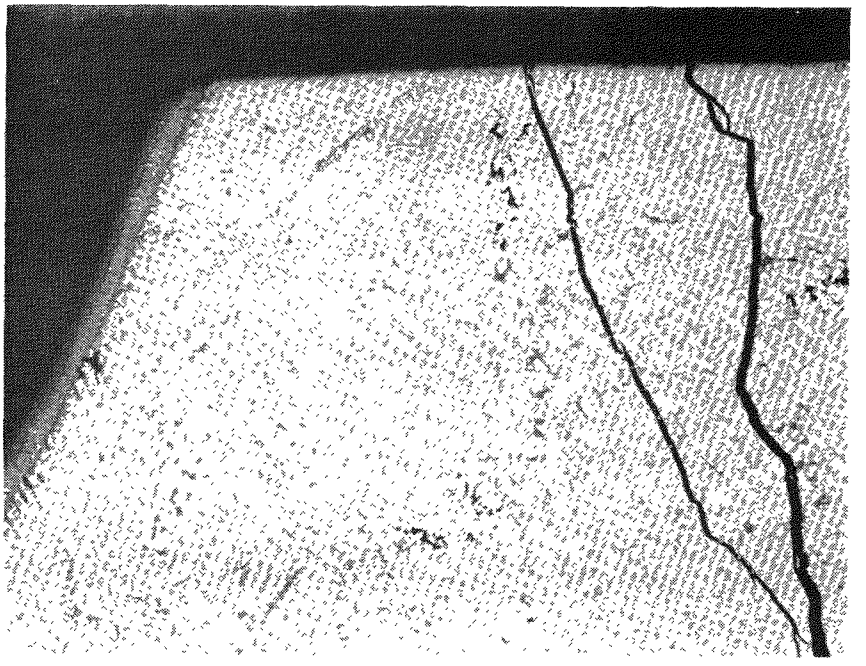

FIGURE 13. Photomicrograph of Weld Zone - Inner Container of M71
125X

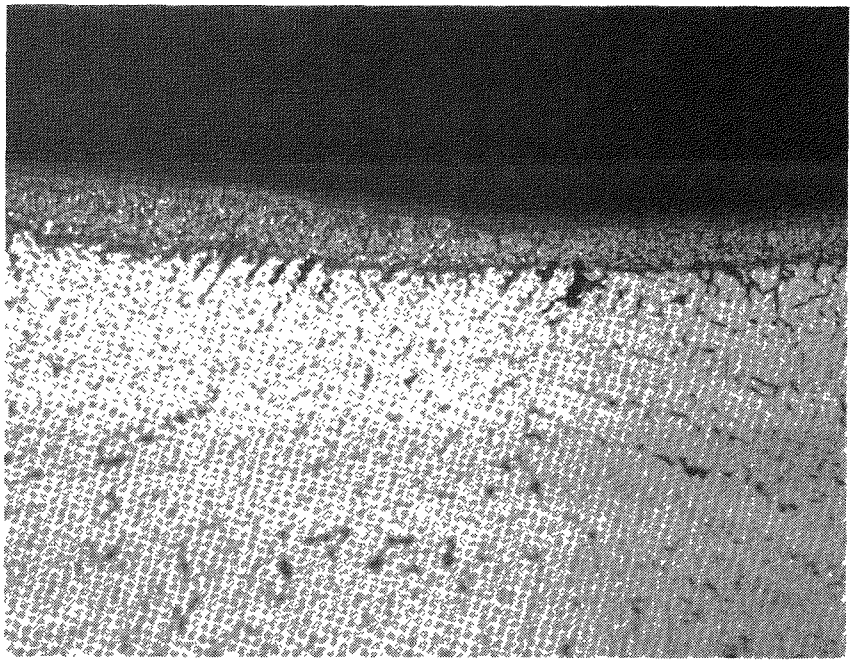

FIGURE 14. Outside Edge of Inner Container of M71
250X

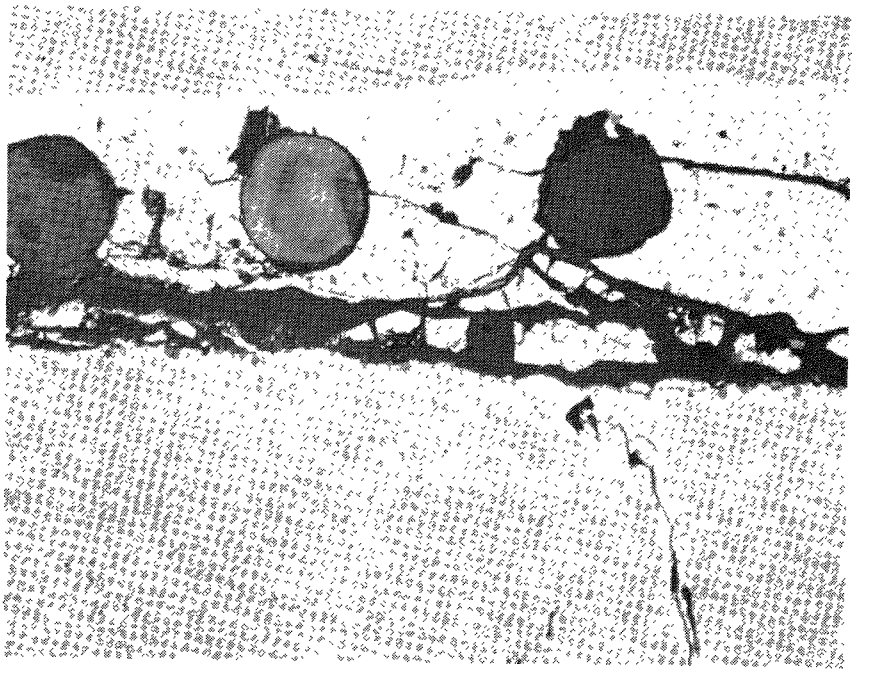

FIGURE 15. Top Lid and Sidewall Interface of 1053S
125X

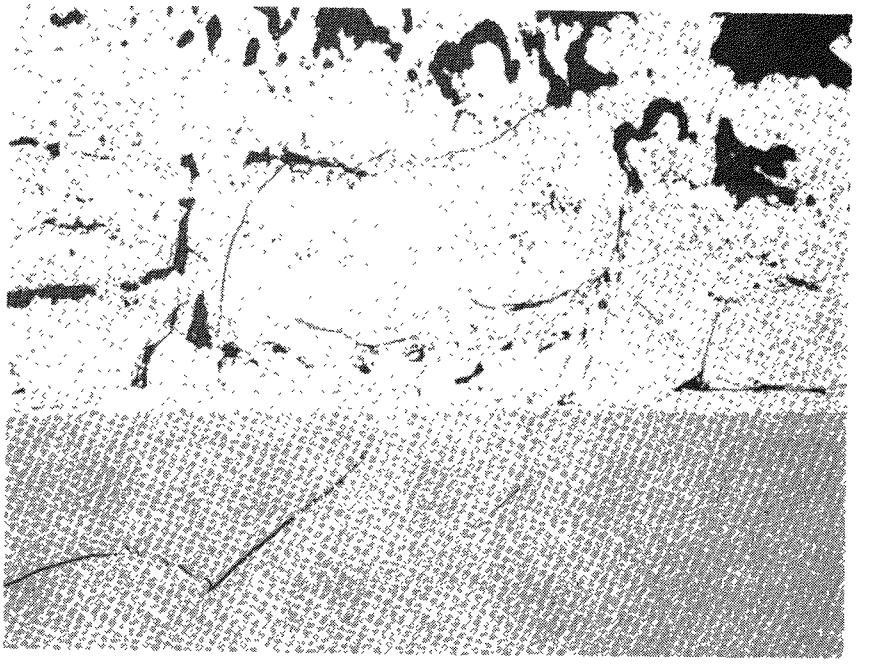

FIGURE 16. Fuel Sidewall Interface Source M71
125X

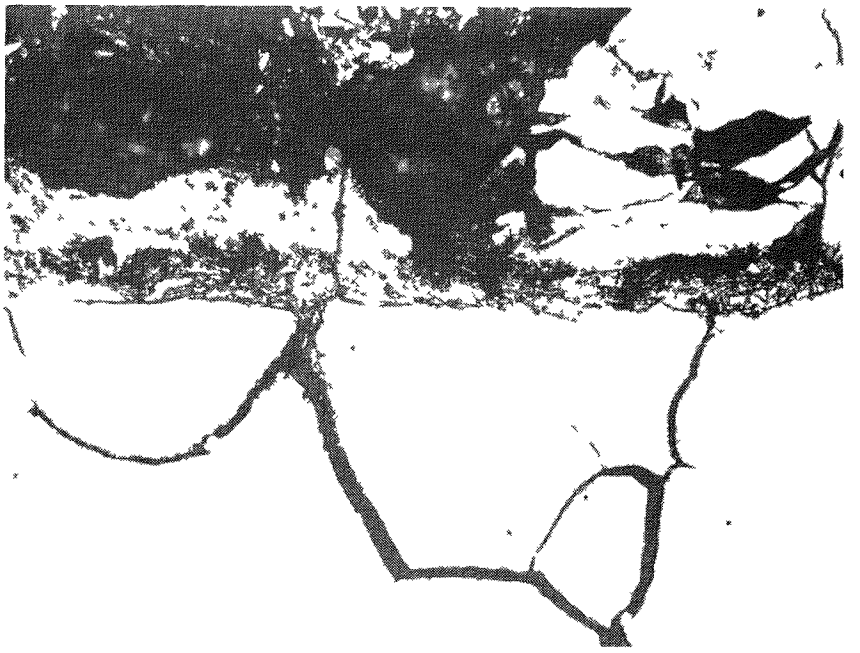

FIGURE 17. Weld Area of Source M71
Inner Container 125X

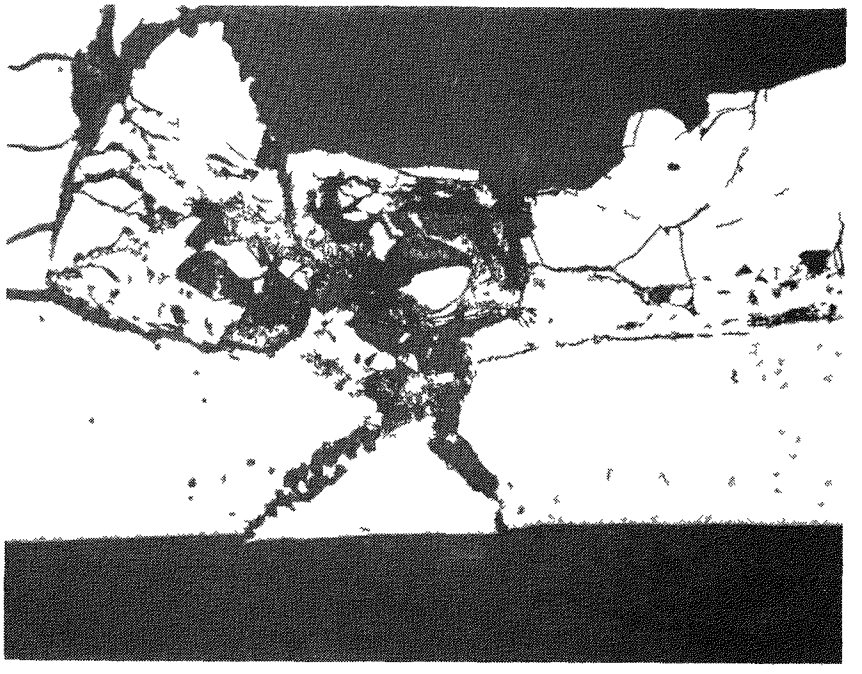

FIGURE 18. Fuel Sidewall Interface M71
125X

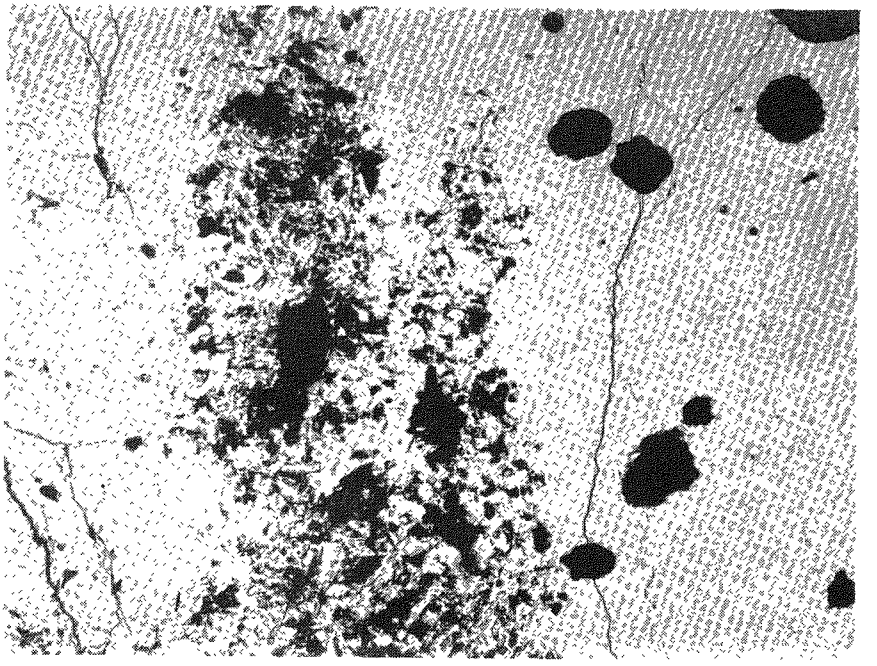

FIGURE 19. Fuel Chunk From 1053S
60.5X

Typical Hardness of SS Outer Container Top Edge and Side Walls

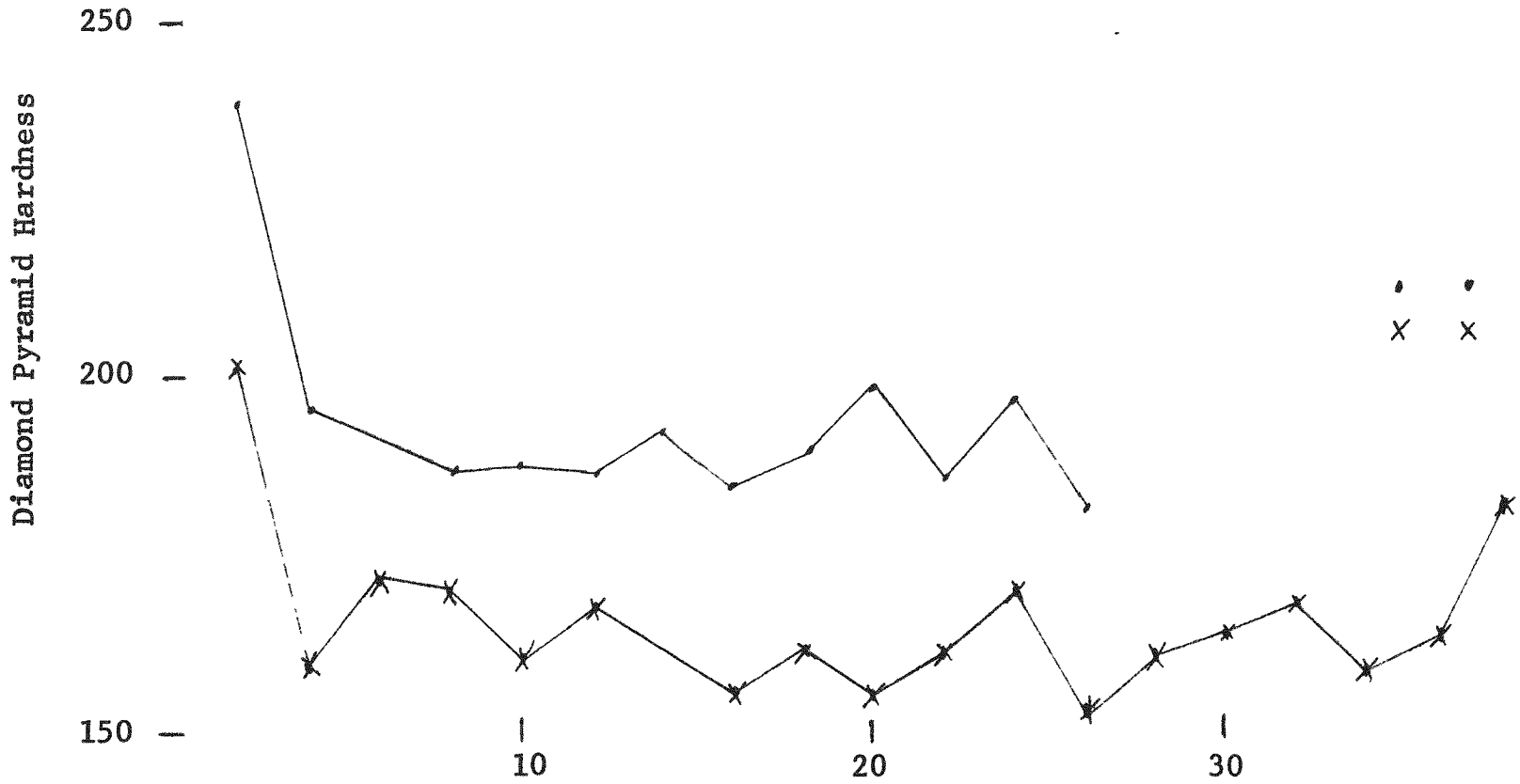

FIGURE 20. Distance from Outside Edge (mils)

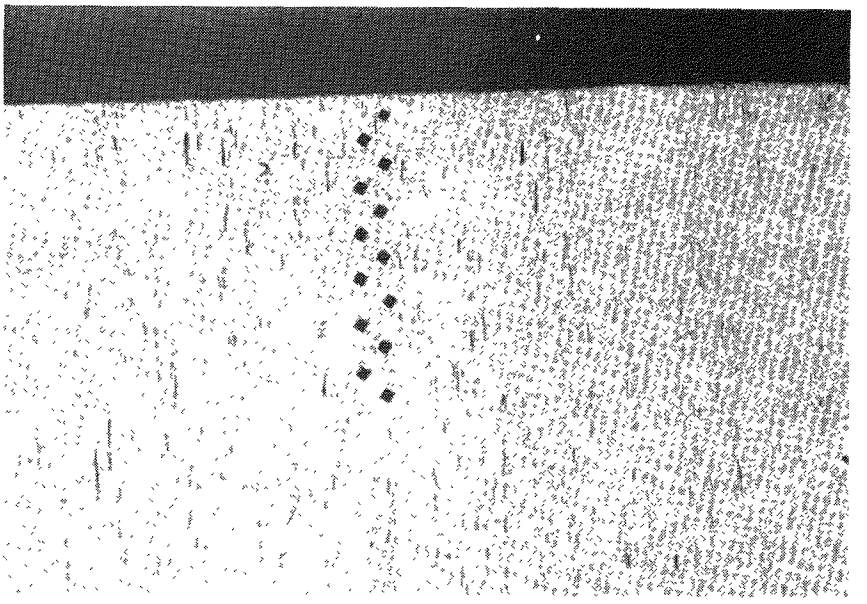

FIGURE 21. Top Edge Source M71 - SS Outer Container
60.5X

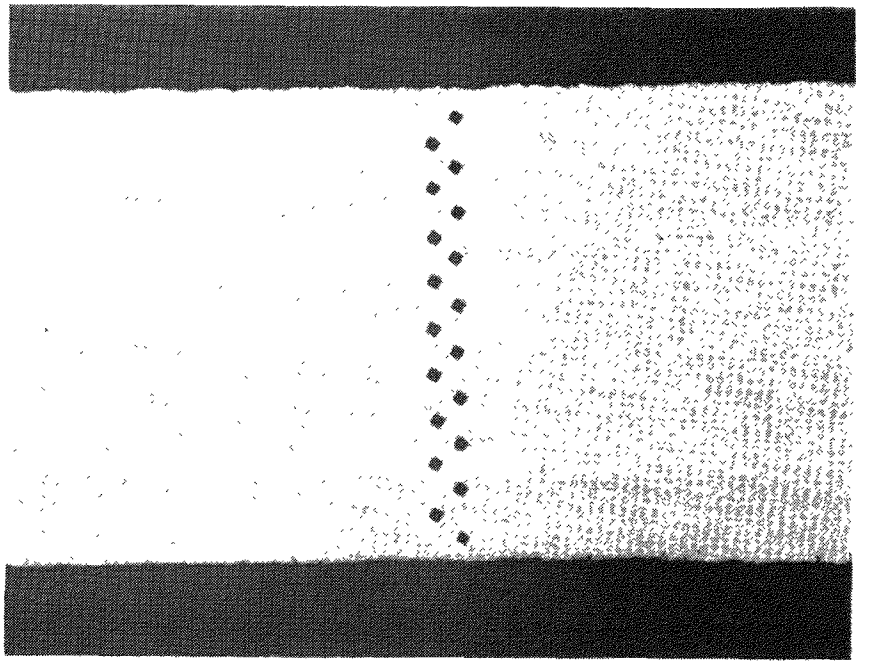

FIGURE 22. Sidewall Source M71 - SS Outer Container
60.5X

Hardness of M-71 SS Outer Top and Side Wall

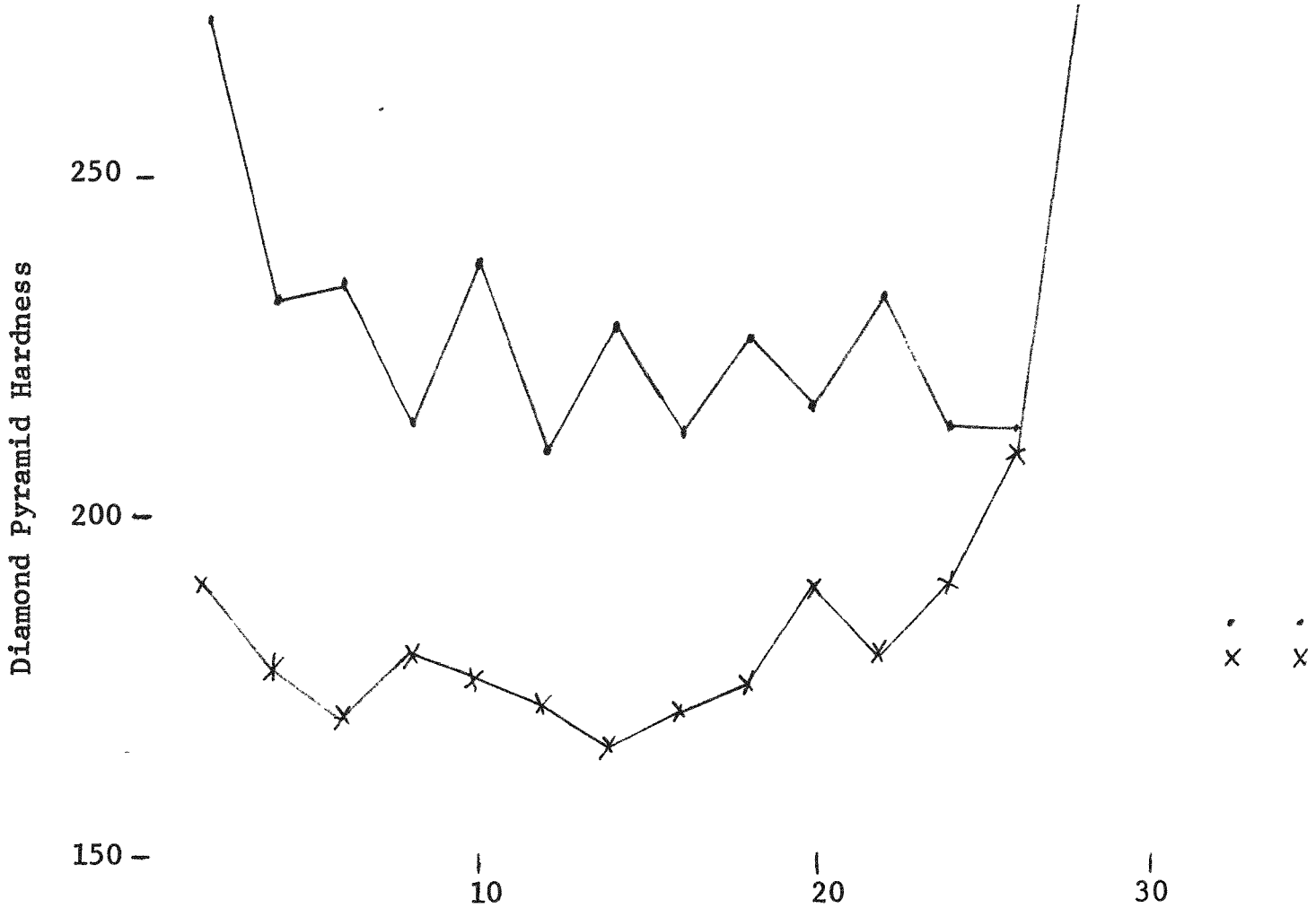

FIGURE 23. Distance from Outside Edge (mils)

APPENDIX A

M-879 - Was returned from the U. S. Air Force, on March 1967, and held in storage. Outer container exhibits flat spots on weld bead, with minimum weld at flat area approximately 0.018", and possible bulge in outer container wall. Inner container exhibits no indication of fuel liner corrosion. Minimum liner weld approximately 0.025"; also bulge in liner wall 0.022", about the middle to the source.

M-259 - Was returned from the USAARDC in January, 1970, and held in storage. Outer container exhibits bulge in outer wall approximately 0.008" long. Outer container weld possibly open. Inner container exhibits no apparent fuel liner corrosion, and no indication of fuel outside the liner; however, liner weld possibly open.

M-472 - Was returned from Gulf L&P Company in April, 1970. Outer liner exhibits no defects. Minimum weld of the outer container is approximately 0.050" long. Inner container exhibits possible fuel liner corrosion, liner weld possible open, with no indication of fuel outside the liner.

M-9 - Was returned from Mobil Research and Development Corp. on February 22, 1971, and held in storage. Outer container exhibits a minimum weld approximately 0.018" long, a slop-fit in end cap, and some bulge in walls. Inner container exhibits no indication of fuel liner corrosion, with liner weld possibly open, and a high density material between liner and outer container at liner weld, possibly fuel.

M-493 - Was returned by Wells Survey Inc. for recanning but inner tantalum container was found unrepairable. Recanned in a single stainless steel container for local handling and transferred to storage. Could have been shipped if an additional outer container was added. Outer container exhibits no indication of defects. Minimum weld approximately 0.025" long. Inner liner exhibits fuel-liner corrosion, liner weld open, and possibly fuel outside the liner.

M-1053-S - Pu-Be Neutron source was never shipped. Defective inner tantalum container. Outer container exhibits no defects. Minimum weld approximately 0.030" long. Inner container exhibits liner wall corrosion. One section of liner completely destroyed. Small amounts of fuel are indicated between liner and outer container walls.

M-71 - Returned from University of California, Radiation Laboratory and was stripped of the outer container July 13, 1963. Tantalum container was badly cracked due to temperatures reached in a short period of time while in a nuclear test. Source unusable, but was recanned in a stainless steel container, and was to be held for further testing. Outer container exhibits no defects. Minimum weld is approximately 0.040" long. Inner container exhibits no apparent fuel liner corrosion. Liner weld joints possibly open.

M-75 - Was returned from Armour Research Foundation of Illinois Institute of Technology. Source subjected to spike heating and was held in storage for inspection of the tantalum shape. Outer container exhibits no defects. Minimum weld approximately 0.025" long. Inner container exhibits possible fuel liner corrosion. Minimum weld approximately 0.025" long.

M-1019 - Was returned from Schlumberger Well Services, Inc. Source enclosed in logging tool and held in shipping container due to high count. Outer container exhibits no defects. Inner container exhibits small amounts of fuel between liner and outer container at weld. Liner possibly open at the weld. Some possible corrosion of the outer surface of the liner at the end cap.

M-1166 - Was returned from Texas Nuclear on December 7, 1970, and transferred to storage. Outer and Inner containers exhibit no defects.

APPENDIX B

Source Number	^{239}Pu (g)	Mfg. Date	Recan Date	Radio-graph	Leak Check	Dose Rate	Neutron Count	Visual & Dimen. Check	Pressure Measure-ment	Destructive Analysis
M-879	160.00	1/31/61	---	X	X	X	X	----	----	----
M-259	79.68	12/4/58	8/24/61	X	---	X	X	X	----	----
M-472	79.87	4/30/59	7/29/63	X	X	X	---	X	----	----
M-9	16.10	11/9/56	8/10/61	X	X	X	X	X	----	----
M-493	79.46	5/20/59	---	X	X	X	---	X	----	----
M-1053S	15.09	1962	---	X	X	X	X	X	X	X
M-71	15.05	10/11/57	7/13/63	X	X	X	X	X	X	X
M-75	15.05	10/17/57	---	X	X	X	X	X	----	----
M-1019	76.34	7/27/61	---	X	---	---	---	---	----	----
M-1166	91.52	10/1/61	---	X	X	X	X	---	----	----