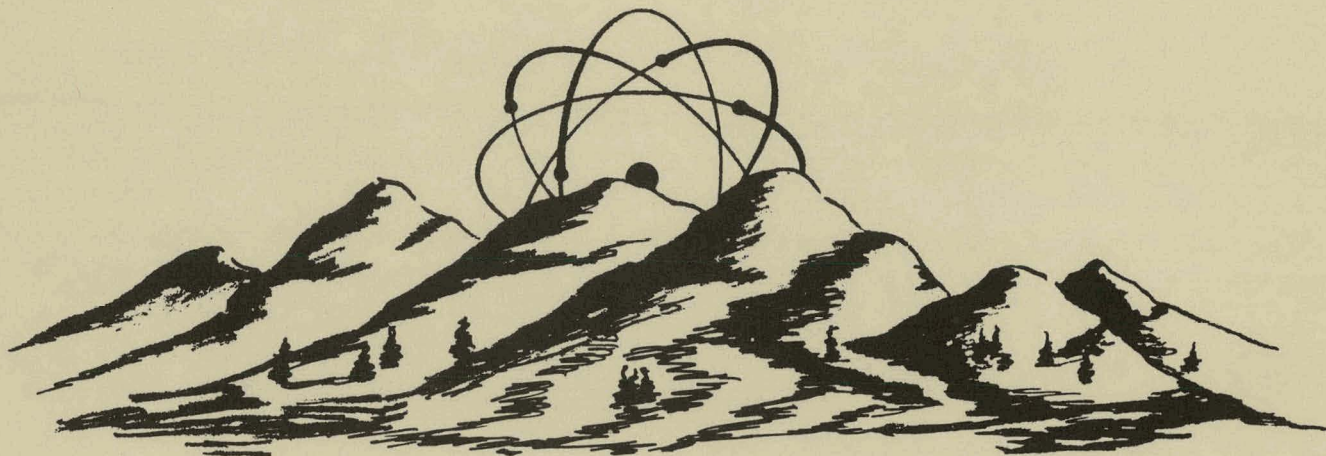


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ISOSTATICALLY PRESSED MAGNESIA STIRRERS  
FOR ELECTROREFINING PLUTONIUM METAL

A. R. Teter



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U.S. ATOMIC ENERGY COMMISSION  
CONTRACT AT(29-1)-1106

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## A B S T R A C T

Several isostatic pressing mold designs were evaluated for fabricating magnesia stirrers. Thin walled neoprene molds produced the most satisfactory one-piece stirrers. The pressed stirrers that were developed have been reused as many as five times in the electrorefining process. Magnesia stirrers formed by suspension casting could be used for only one electrorefining run.

## ACKNOWLEDGMENTS

The author is indebted to W. P. Meier for his assistance in fabricating and evaluating the molds during this study. He is also indebted to D. J. Kessel Ceramics Group supervisor for his technical advice during the project and aid in preparing this report.

## INTRODUCTION

Magnesia stirrers are used to agitate the plutonium metal and the electrolyte during an electrorefining process. High purity plutonium metal is separated from impurities by setting up an electric potential between a cathode and anode within a molten salt solution of alkali and plutonium chlorides. Very pure magnesia hardware is used in the electrorefining process because that hardware is resistant to chemical attack from molten plutonium and molten salt. The stirrers used in this process must withstand high temperatures, thermal shock, abrasion, and chemical attack for long periods of time.

Previously, magnesia stirrers (Figure 1) were cast in plaster molds by using a powder and water suspension which is called slip. The blades and the shafts were cast in separate molds. After the parts were cast they were glued together with a thick magnesia paste and then fired. Approximately 60 percent of the slip-cast stirrers were unsatisfactory. The following factors contributed to the high loss:

1. Breakage during removal from the complicated plaster molds.
2. Voids which were caused by air trapped during casting.
3. Voids between the blade and the shaft resulting from poor gluing.

Isostatic pressing was investigated as a forming method because the processing variables can be controlled more readily than in slip casting. With this forming method, the prepared powders are sealed in a flexible mold and pressed in a hydrostatic pressure vessel. It was believed that by isostatically pressing a monolithic stirrer the void problem and the gluing problem would be eliminated.

## PROCEDURE

Three different isostatic pressing molds were evaluated in this study. The molds were designed oversize to allow for 25 percent pressing compaction and 18 percent linear

firing shrinkage. The first mold was made from polyvinyl chloride plastic and was designed to press a paddle-shaped stirrer (Figure 2). Polyvinyl chloride is flexible and distorts easily during processing; however, this mold was held rigid, during filling, with magnesia powder and pressing with a support container. The second mold (Figure 3) was dip formed with neoprene rubber and was also designed to press a paddle-shaped stirrer.

A third mold was designed to press a stirrer (Figure 4) similar to the slip-cast stirrers. The steel model used to form this mold has removable blades that fit in slots in the shaft. The blades were made removable so that the dipped neoprene pressing bags could be easily removed from the model. The support container for this mold is slotted to accommodate the movement of the mold during pressing. Without these slots the blades would be sheared from the shaft during pressing.

The magnesia powders\* used for pressing the stirrers were prepared by ball milling and spray drying. These powders contained 4.0 percent polyvinyl alcohol as a temporary binder and 3.0 percent yttrium oxide as a sintering aid. Spray dried powders have a uniform coating of binder on each particle, are spherical in shape, and are therefore free flowing. The spray dried powders had an average particle size of 20 microns.

The prepared powders were poured into the molds and isostatically pressed at 20,000 psi. This pressure was held for 60 seconds and released at a uniform rate in approximately 30 seconds. An air-operated device (Figure 5) was fabricated to remove the stirrers from the mold. The large end of the mold is clamped to the receiver and air pressure is applied to balloon the mold, allowing the stirrer to slide out.

The pressed stirrers are fired in the horizontal position while bedded in -10 +20 mesh magnesia grain. The temporary binders are burned out at a rate of 50°C per hour between 300 and 500°C. The stirrers are then

\*Norton Refractories Company -200 mesh arc fused magnesia.

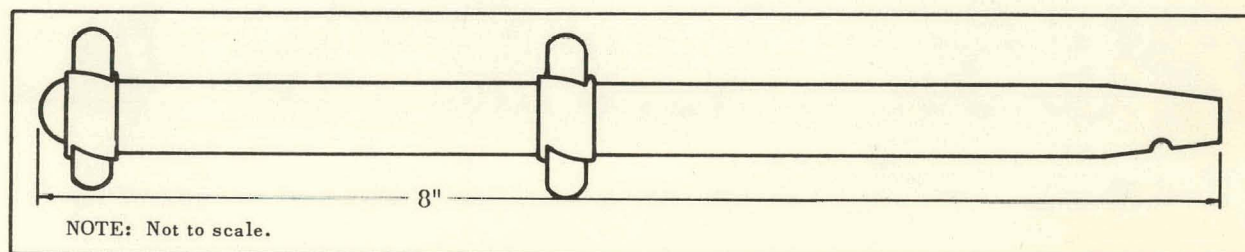


Figure 1. SLIP CAST MAGNESIA STIRRER



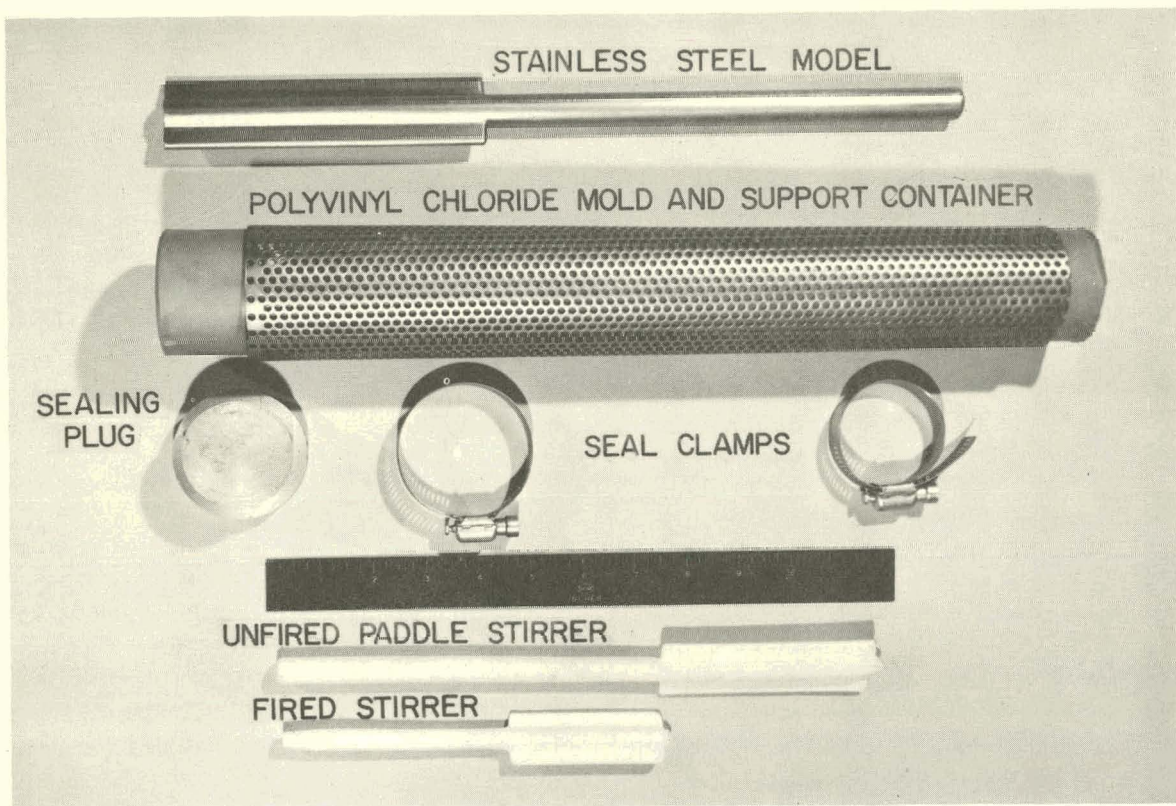


Figure 2. POLYVINYL CHLORIDE MOLD FOR PADDLE STIRRER

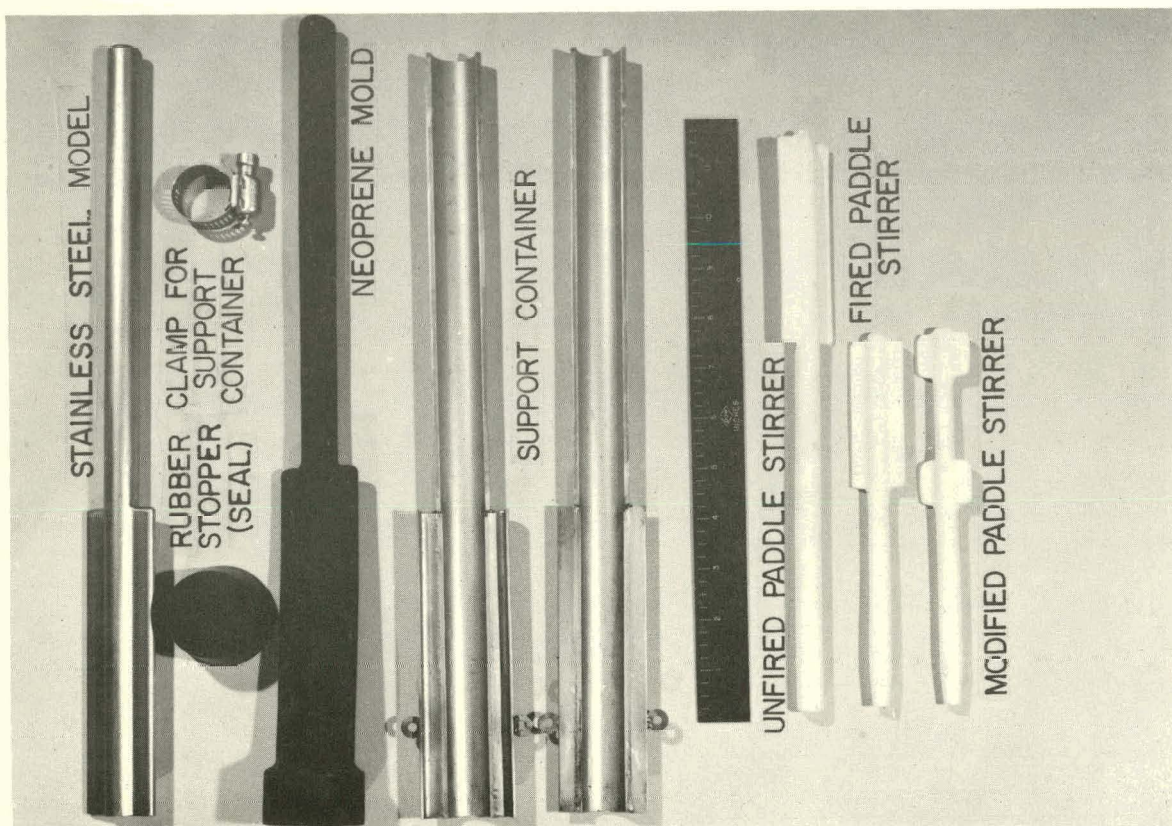


Figure 3. NEOPRENE MOLD FOR PADDLE STIRRER



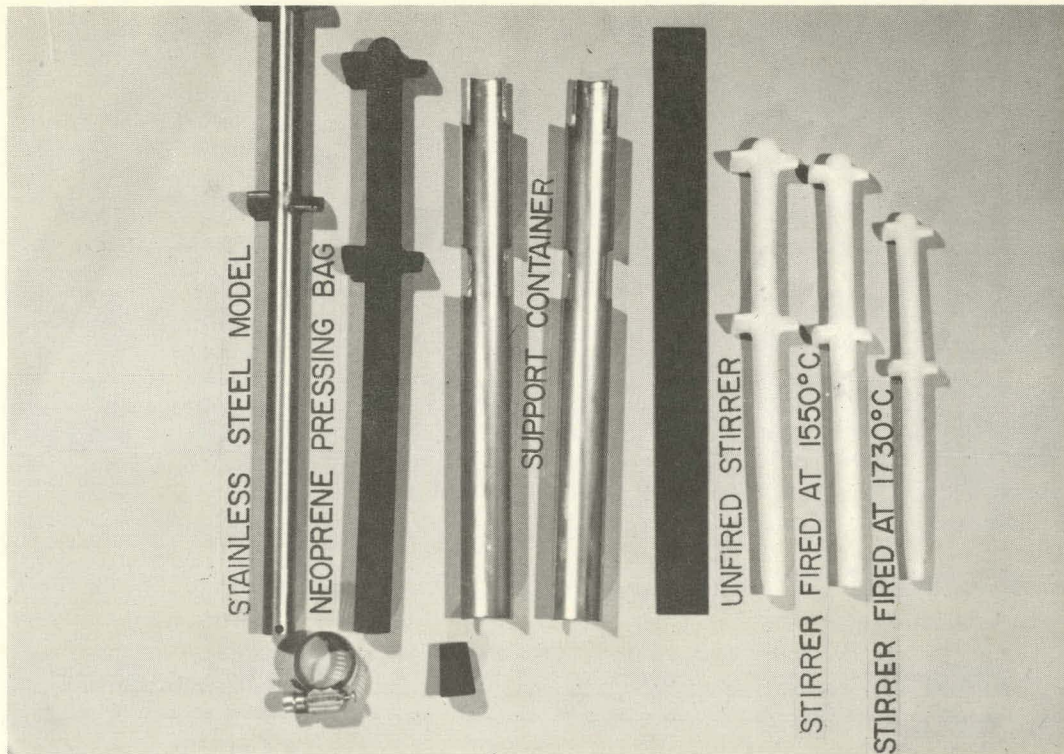


Figure 4. TWO-BLADED STIRRER MOLD

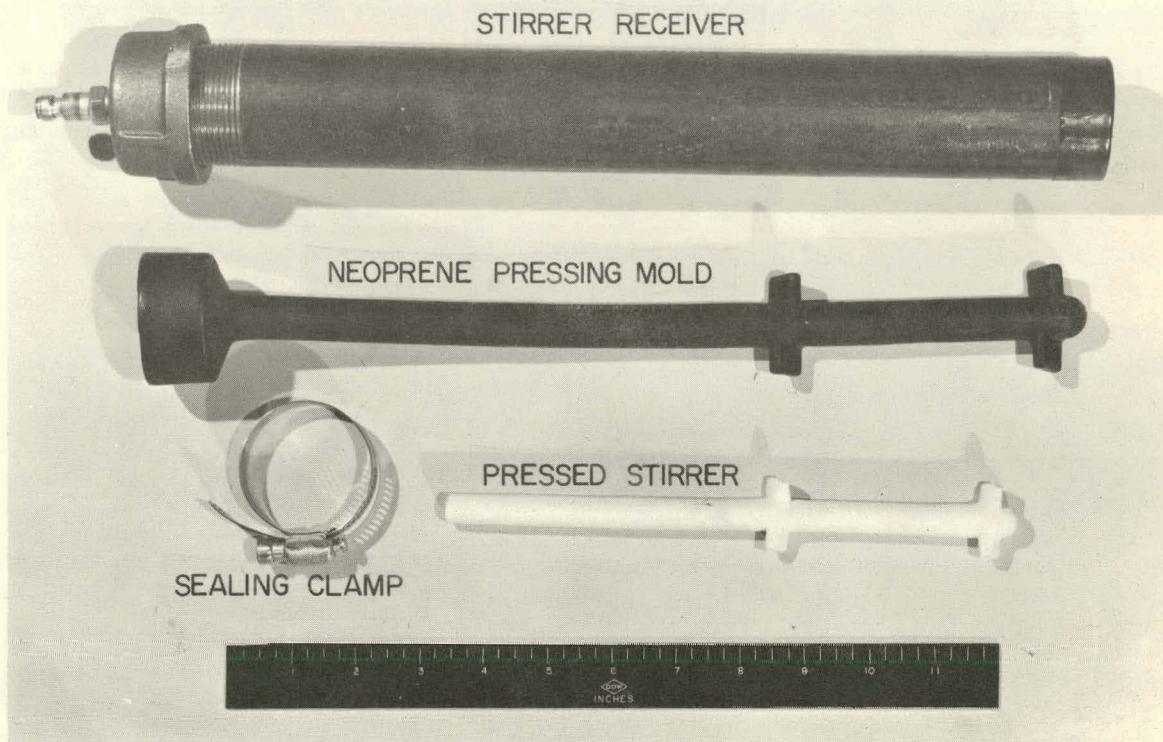


Figure 5. STIRRER REMOVAL DEVICE



sintered at 1730°C for 8 hours. The tapers for attaching the stirrers to the mixer chuck are diamond ground after firing.

## RESULTS

The polyvinyl chloride mold (Figure 2) broke the stirrers during decompression of the isostatic press because of the spring-back of the polyvinyl chloride. The mold was modified so that the ends were held rigidly by placing clamps between the end seals and the perforated container. Pressure was then applied, and relieved, perpendicular to the axis of the shaft. It was also necessary to use a mold lubricant, such as melted paraffin, to successfully press stirrers with this mold.

More than 100 stirring paddles have been pressed with the neoprene mold shown in Figure 3. A modification was necessary on these stirrers to prevent excessive splashing during electrorefining. The modified paddle stirrer shown at the far right of Figure 3 is made by grinding the central web from a fired paddle stirrer.

Approximately 50 stirrers have been fabricated with the two-bladed stirrer molds (Figure 4). The mold removal device will remove the stirrers from the molds, but the molds are badly deformed and cannot be reused again. We are presently cutting the molds from the pressed stirrers with an X-Acto knife.

Very few stirrers are lost during the isostatic pressing operation. Approximately 20 percent of the stirrer shafts warp during firing at 1730°C.

## DISCUSSION OF RESULTS

Fabrication costs for slip casting and isostatic pressing the stirrers are shown in Table I. The pressed stirrers were fabricated for one-fifth the cost of the slip-cast stirrers. The slip-cast stirrers were suitable for only one refining run, while pressed stirrers have been used for up to five refining runs. Plutonium enters into large voids in the glued areas of the slip-cast stirrers and breaks them because its thermal expansion is greater than that of magnesia. Isostatic pressing produces stirrers that are free of large voids.

TABLE I

FABRICATION COSTS OF MAGNESIUM OXIDE STIRRERS

	<u>Slip Casting*</u>	<u>Isostatic Pressing*</u>
Molds	\$ 15.00	\$ 4.00
Magnesia		
Preparation	2.00	5.00
Forming	100.00	9.00
Firing	5.00	4.00
Finishing	3.00	3.00
TOTAL (each)	\$125.00	\$25.00

\*Cost based on pilot production.

Two-bladed stirrers were found to be the most suitable stirrer for use in electrorefining. However, the modified paddle stirrer is a suitable substitute. The central web must be diamond ground from the modified paddle stirrer, making it more expensive to produce.

The neoprene molds for the two-bladed stirrer are deformed when they are removed from the pressed stirrer with the mold removal device. It is believed that the two-bladed stirrer molds could be reused if they were fabricated from latex rubber. The literature indicates that latex rubber is more resistant to permanent deformation than neoprene.

The stirrers used in the electrorefining process must be straight because they are rotated at 1500 rpm. Approximately 20 percent of the stirrers warp during firing and are not suitable for use in electrorefining. To prevent warpage, it is believed that the stirrers must be suspended vertically during firing. Preliminary studies with vertical firing has indicated that an 8-hour soak at 1730°C does not improve the fired straightness. It is believed that a 20-30 hour soak will be required at 1730°C before enough plastic flow would occur to improve the straightness.

## SUMMARY

Monolithic magnesia stirrers were fabricated by isostatic pressing. Isostatic pressing was found to be a less costly, more reliable method than slip-casting for fabricating magnesia stirrers.