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THE SLICING METHOD FOR MAKING HARP

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THE SLICING METHOD FOR MAKING HARP

H. A. Tanner and R. L. Stetson

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Approved by:

Dr. Peter King, Head, Polymer Section
Dr. P. Borgstrom, Superintendent, Chemistry Division

Problem No. 32R-06-01 March 9, 1948

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ABSTRACT

A method of producing Harp material by slicing a molded cake is described. Equipment design, operating procedures, formulations, and results are given. About 60 square yards of Harp have been made at the Laboratory by this method.

PROBLEM STATUS

This is an interim report on this problem; work is continuing.

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THE SLICING METHOD FOR MAKING HARP

INTRODUCTION

Present requirements of the service for Harp material for use in the 600-Mc and X-band regions indicate the need for a method of fabrication which would allow a relatively high rate of production of this material.

The wavelength to be absorbed and the composition determine the thickness of the absorber. Functional requirements and the bandwidth of absorption limit the tolerance on thickness. For the 600-Mc and X-band wavelengths and with the pigments currently available the thickness will range from 50 mils to 400 mils, maximum desirable tolerance being 2 percent. The material must be nondirectional, weather and shock resistant, and somewhat flexible. These facts impose severe restrictions as to possible manufacturing procedures.

During the war the DuPont Experimental Station, working successively under contracts N-173s-5822, OEMsr-1199, and N5-ori-17, and in cooperation with the M.I.T. Radiation Laboratory Projects NA-131 and NS-296 under contract OEMsr-262, investigated the so-called "slicing method" of producing Harp films. Their results, summarized in 3 DuPont Reports*, were so promising that it seemed advisable to continue along that line. The "slicing method", standard for producing heavy sheets of celluloid and certain other plastics, consists of molding a massive cake of material and slicing off film of the required thickness.

*Following Reports from the DuPont Chemical Department Experimental Station:

G. T. Borcherdt, B. C. Pratt, and C. W. Theobald, "Special Protective Coatings XXII - Preparation of Film by Hot Pressing," Report ESP-45-234

B. C. Pratt, "Special Protective Coatings XXV - Final report of all work under OSRD Contract OEMsr-1199", Report ESP-45-237, 1 December, 1943 - 30 September 1945

J. H. Bladt and C. W. Theobald, "Special protective coatings - process studies and training of NRL personnel - final report," ESP-46-41

[redacted]

Harp material can also be made by the spraying, cross-knifing, or hot-pressing methods. The spraying process deposits material in coats about 1 mil thick with forced drying between coats. For thicknesses exceeding 50 mils, the process becomes impractical because of the number of coats and the time required for solvent to be removed from thick films. The same disadvantage holds true for the cross-knifing process which consists of spreading thin films, from solution, drying, spreading on a second film cross-wise on the first, etc,, until the required thickness is built up. The hot-pressing method is similar to the slicing method, except the material is molded directly to size, instead of molding a large cake and slicing to size. The hot-pressing method saves no time over the slicing method and it is hard to obtain correct and uniform thicknesses, therefore, the yield of good material is less than for the slicing method.

EQUIPMENT

Presses and standard compounding equipment including rubber mill, Banbury mixer, and calender were already available at the Laboratory. Other items needed were a mold and a slicing device. The needs of the Laboratory could most readily be met by adapting a metal planer in the machine shop for the slicing operation.

A 66-inch hydraulic-drive open-side shaper planer was selected having a 24-inch wide planer bed. The hydraulic drive is desirable to prevent chattering. An adjustable knife blade holder (Figure 1 - part of NRL Drawing F 1053A) for a 19-inch knife was devised for the tool post. The blade is stationary during slicing and the cake is pulled under it on the planer bed. Thickness of slice is regulated by vertical adjustment of the knife holder.

A knock-down-type mold was designed. This construction has three advantages; no single part is cumbersomely heavy, the block of material is easily removed after molding, and the inner base plate with the block of material molded fast can be bolted directly to the planer bed. The size of cake to be molded was determined by the width of the planer bed, the size press available, and the necessity of curing uniformly through the cake. Two molds, one for a cake 18 x 24 x 2 inches and the other for a cake 18 x 24 x 4 inches were eventually constructed. Assembly drawings of the molds are shown in Figures 2 and 3 (parts of NRL Drawing F 1053). The molds were designed to withstand 2000 psi internal molding pressure.

The molds and blade holder were designed by the Engineering Services Division of NRL from general specifications by the Chemistry Division. Plastic slicing equipment used at DuPont was investigated.

FORMULATION STUDIES

The physical properties desired in the product dictate the use of a vulcanized rubber binder. In previous work at this Laboratory it was found

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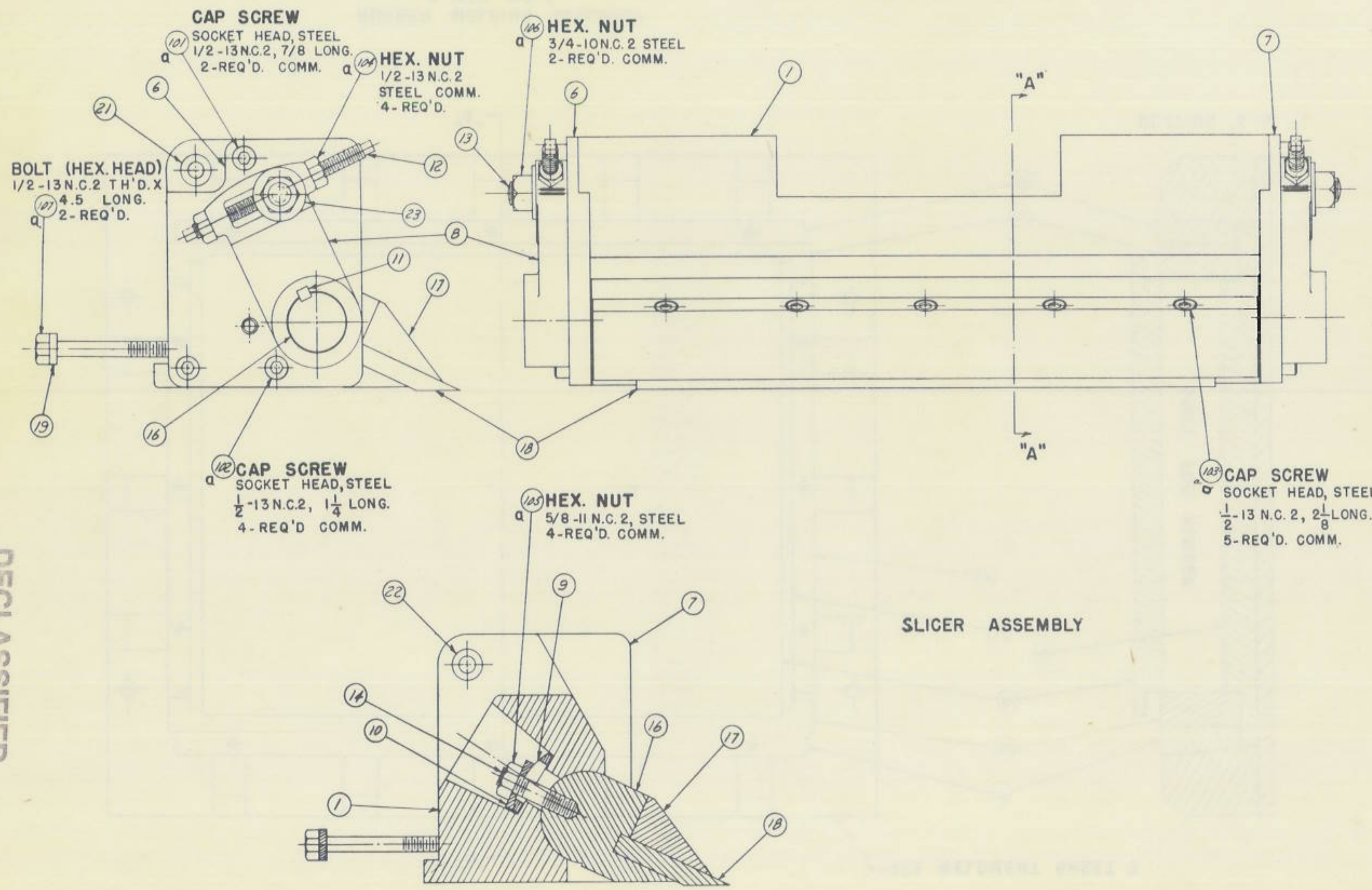

Fig. 1 - Rubber Mold and Slicer Assembly - Scale 3/16" = 1"

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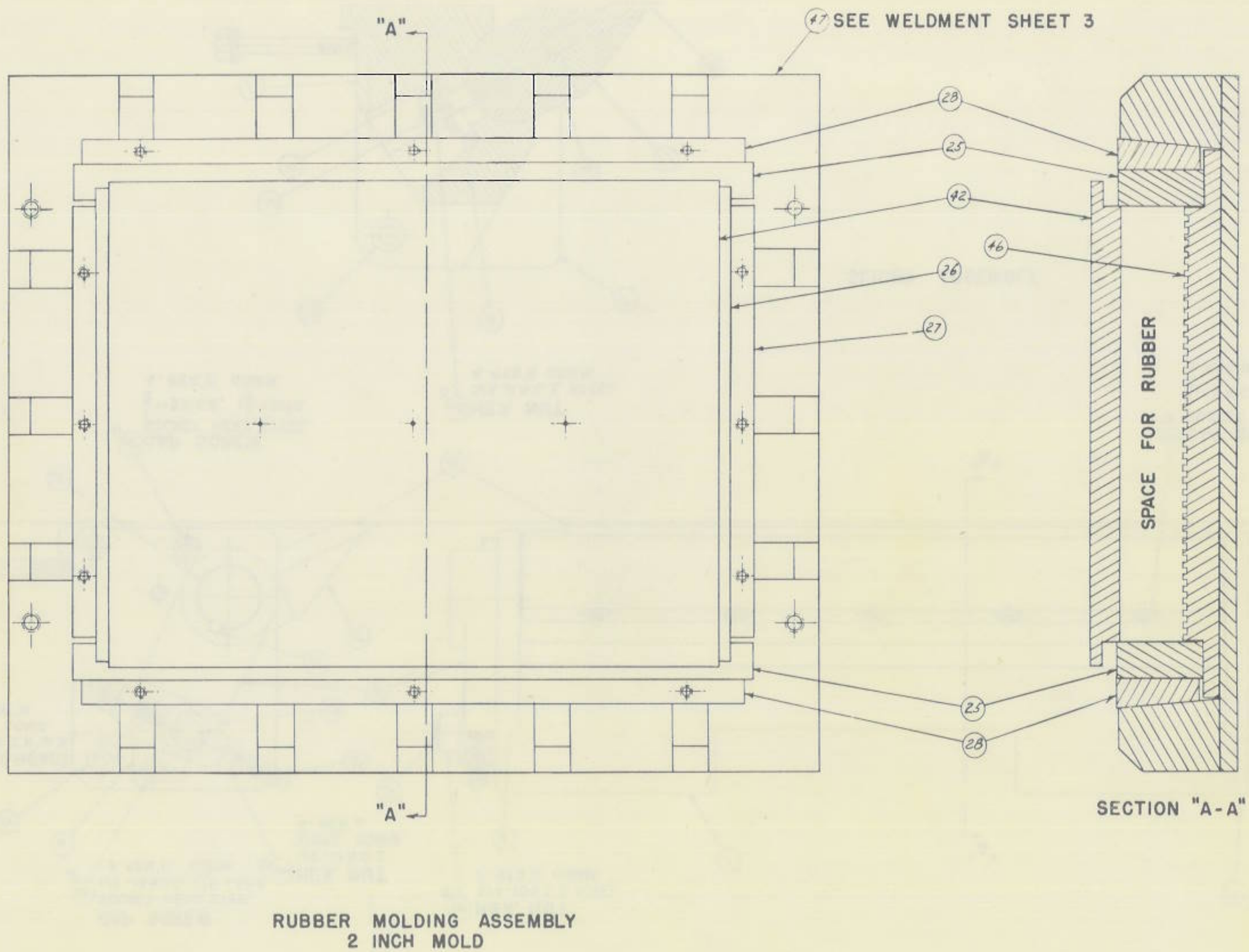

Fig. 2 - Rubber Mold and Slicer Assembly - Scale 1/8" = 1"

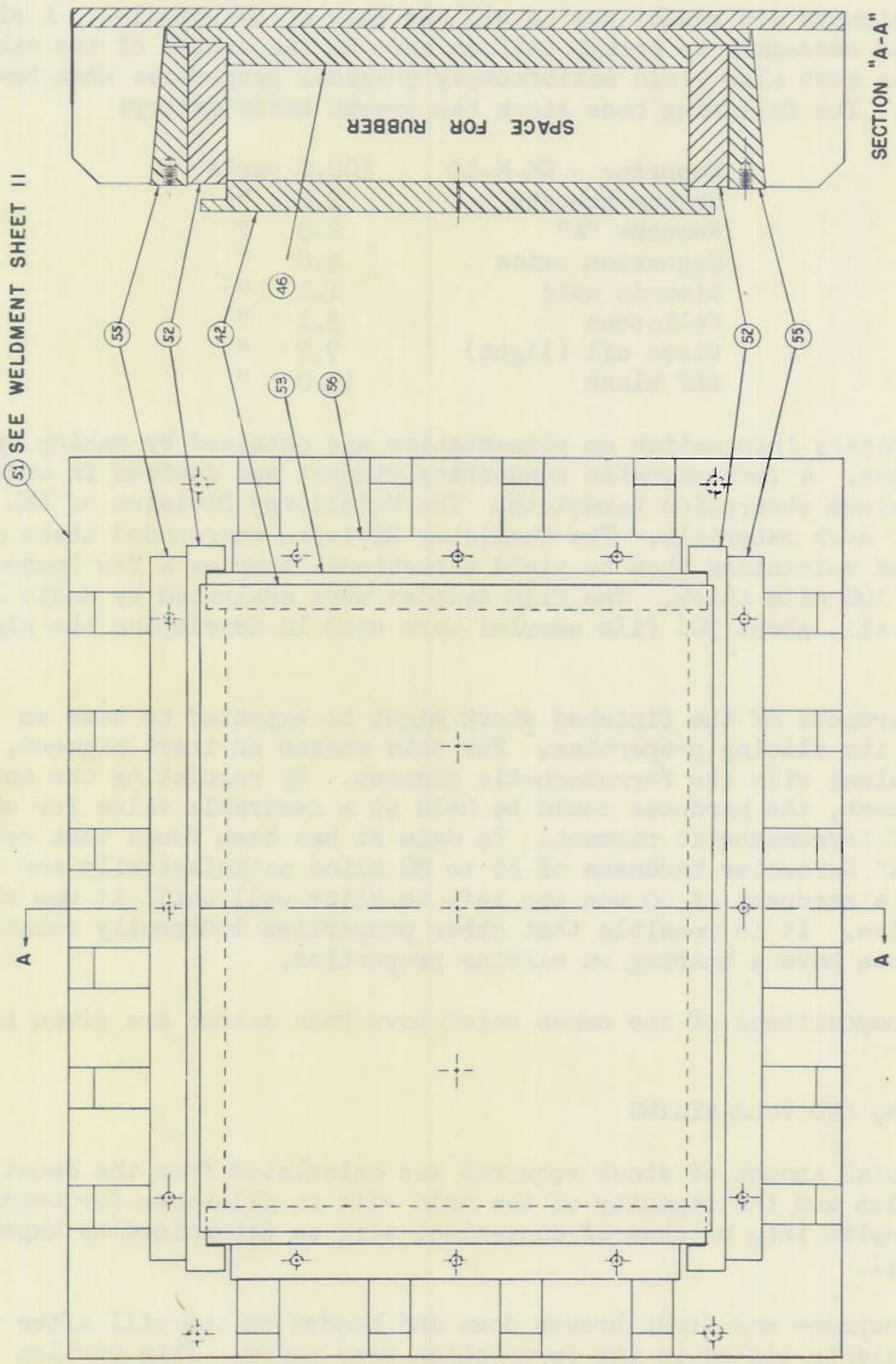

Fig. 3 - Rubber Mold and Slicer Assembly - Scale 1/8" = 1"

that sulphur vulcanization adversely affected some iron pigments; accordingly a neoprene binder was selected. Additional advantages of neoprene over other elastomers are resistance to oil and ease of compounding. A slow curing rate is necessary to permit uniform cure to the center of the cake. The composition must also yield satisfactory physical properties when heavily pigmented. The following base stock has proven satisfactory:

Neoprene - GR-M-10	100.0	parts
Sodium acetate	2.0	"
Neozone "A"	2.0	"
Magnesium oxide	4.0	"
Stearic acid	1.1	"
Heliozone	5.1	"
Circo oil (light)	7.7	"
SRF black	19.0	"

Preliminary information on pigmentation was obtained by making up small film samples. A ferromagnetic conducting pigment was desired in order to obtain maximum absorption bandwidth. The Metallurgy Division of NRL supplied samples of such materials. The Chemistry Division compounded these and calendered and vulcanized them to yield directional samples a few inches square and 50 to 100 mils thick. The film samples were evaluated by Radio I Division. In all, about 300 film samples were used in developing the pigmentation.

The hardness of the finished stock might be expected to have an important effect on its slicing properties. For this reason an inert pigment, clay, was used along with the ferromagnetic pigment. By regulating the amount of inert pigment, the hardness could be held at a desirable value for changing amounts of ferromagnetic pigment. To date it has been found that cakes with a Shore "A" Durometer hardness of 65 to 80 slice satisfactorily and that a cake with a hardness of 40 was too soft to slice well until it was chilled with dry ice. It is possible that other properties indirectly related to the hardness have a bearing on slicing properties.

The compositions of the cakes which have been sliced are given in Table I.

COMPOUNDING AND VULCANIZING

The total amount of stock required was calculated from the density of test samples and the capacity of the mold with an allowance for overflow. This was split into batches of convenient size as determined by experience on the mill.

The neoprene was first broken down and banded on the mill after which the ingredients listed in the formulation were added. This portion of the operation took about 15 minutes. If clay was used as a filler, it was added at this point, requiring another 10-15 minutes. The mill was then closed tight and the stock passed through 3 to 4 times in order to thoroughly disperse all ingredients. Finally, the ferromagnetic pigment was added, usually requiring 15-20 minutes. The stock was then cut across six times and passed through six times with the mill set at on opening of approximately 30 mils.

TABLE I

Compositions and Characteristics of Cakes Which Have Been Sliced

Cake No.	Base Stock (%)	Ferromagnetic Flake (%)	Clay (%)	Vulcanizing Time (hr)	Vulcanizing Temp. (°F)	Shore "A" Hardness	Slicing Properties
1	33 1/3	66 2/3	0	14	300	67-70	Good
2	33 1/3	66 2/3	0	14	300	67-70	Good
3	33 1/3	66 2/3	0	14	300	67-70	Good
4	66 2/3	33 1/3	0	14	300	40+	Poor
5	44.4	31	24.6	14	300	70-73	Good
6	45	30	25	14	300	70-72	Good
7*	45	28	27	16	300	70-73	Good
8	45	28	27	14	300	70-73	Good
9	47	22	31	14	300	75-78	Good

* Cake 7 was a 4-inch cake; all others were 2-inch cakes.
\+ Cake 4 when chilled with dry ice had a hardness of 55 and then gave satisfactory slicing properties.

Comparative tests were run with the rubber mill versus a Banbury mixer of approximately the same capacity, special note being made of the labor cost and material loss inherent in each process. The rubber mill seemed to be superior to the Banbury Mixer in quality of dispersion and with respect to material loss. The net material loss for the complete compounding procedure on the rubber mill averaged about 0.2% and the loss on the Banbury Mixer was over 1%.

In preparing the mold for vulcanization, the base plate was given a thin coat of adhesive to hold the molded cake. Ty-ply "S" a well-known vulcanizing adhesive for neoprene, was used. The slides and top of the mold were heated and coated with a silicone mold release (Dow-Corning Silicone Mold Release No. 35). The base plate and slides were then assembled ready to receive the stock. Finally, two sheets of 18 x 24 inch aluminum foil of 2-mil thickness were heated and coated with silicone mold release. These were placed on top of the cake under the top plate to prevent the adhesion of vulcanized rubber to the top plate.

The accepted method for filling a mold with raw stock prior to vulcanization is to calender the stock into sheets and pack it tightly into the mold, removing as much occluded air as possible in the process. Such a step immediately introduces the problem of directionality in the finished

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product, i,e., the calendering operation has a tendency to align any particles dispersed in the rubber. This effect carries through the vulcanizing step and adversely affects the electrical characteristics of the HARP material.

To prevent directionality, the stock was calendered at a thickness approximately one-fifth the predicted thickness of the finished sheets. The slab was then built up uniformly in layers, the direction of calendering in each layer being at 90° to that of the sheets in adjacent layers. Each layer was trimmed to slightly less than 18 x 24-inches. In order to reduce trimming and fitting of the stock to a minimum, overlapping within layers was permitted. Such overlaps were kept as near the center of the slab as possible, thereby providing that all flow would be outward when pressure was applied in the vulcanization process.

Preforming of the slab was accomplished with the mold in the press. The temperature was held constant at about 160° F for one to one and one-half hours and pressure applied slowly, commencing at about 150 tons and gradually increasing to a maximum of 300 tons. During this time, pressure was intermittently reduced to zero to break down the stock and remove all occluded air. After the pressure had been increased to 300 tons, the temperature was raised to 300° F and vulcanization carried out at this temperature for 14 hours. The mold was then cooled and disassembled.

In an attempt to eliminate the directionality introduced by calendering, preformation of one cake was accomplished by passing all stock through the Banbury mixer and placing it directly in the mold. The result was the occlusion of a considerable amount of air in the vulcanized slab; therefore, this modification of the procedure was discarded.

SLICING

The inner base plate of the mold with the attached cake was bolted to the bed plate of the planer with the long dimension in the direction of travel. A wood block was braced against the rear end of the cake to prevent distortion during the last part of the cut. The blade was allowed to enter the wood slightly at the end of the cut.

The blade was honed for each cake. Originally it was planned to purchase the blade from an outside source, but because of nondelivery, a blade having a Rockwell "C" hardness of about 40 was made at the Laboratory and functioned satisfactorily. It is possible that a harder blade would be better. In operation it has been found that the best results were obtained by adjusting the blade to make the smallest practical angle with the direction of travel. No lubricant is used on the blade.

The clearance of the blade above the base plate was adjusted to take a thin leveling cut from the top of the cake. It was then lowered to cut off a slice having the thickness indicated by test results on the preliminary or formulation samples. This first slice was then tested and the remainder of the cake sliced to the thickness indicated by this test. Slices ranging from 29 to 350 mils have been cut.

QUALITY OF PRODUCT

With a cake properly made up to be free of bubbles, the thickness of a slice will not vary more than 2 mils and variations between different slices will be less than 2 mils. Under the same conditions, the electrical properties are uniform and directionality has been reduced to a minimum.

The following table gives test results on a sample of cake No. 9. The bandwidth of absorption on this cake was not as great as that of others, however, these results are used to show more effectively the degree of directionality to be expected.

TABLE II

Test Results on 71-mil Sample of Cake No. 9 (Readings Taken with E-Vector Perpendicular to Plane of Sample)

Wavelength (CM)	% Reflection	
	First Direction*	Second Direction*
3.16	3.0	6.0
3.31	0.4	1.2
3.41	0.2	0.2
3.53	1.5	0.3
3.73	6.0	3.2
3.89	11.0	8.0

* Sample rotated 90° between these readings

There was no variation in hardness or electrical properties of slices sampled through the cake. With the exception that the surface had a "cut" or unpolished appearance there seemed to be little difference in physical or electrical properties between slices and molded slabs of the same composition.

To date about 60 square yards of Harp material have been made at NRL by the slicing method. It is believed that once the procedure has been adapted to a given composition, a yield of about 90 percent usable material could be expected.

CONCLUSION

This slicing method is satisfactory for producing Harp when the thickness will be 50 to 400 mils.