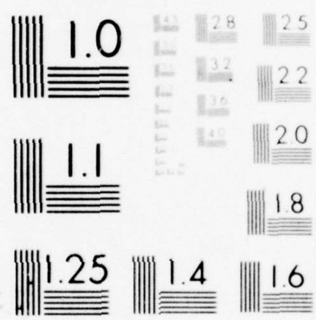
AD-A034 794 RAYTHEON CO WALTHAM MASS RESEARCH DIV F/G 9/5
MANUFACTURING METHODS AND TECHNOLOGY MEASURE FOR ARC PLASMA SPR--ETC(U)
OCT 76 H J VAN HOOK, D MASSE, J SAUNDERS DAAB07-75-C-0043
UNCLASSIFIED S-2112 NL

1 OF 1
AD
A034794

END
DATE
FILMED
2-77


1.0
2.8
2.5
3.2
2.2
3.6
4.0
2.0
1.1
1.8
1.25
1.4
1.6

(12)

FG.

Fifth Quarterly Progress Report

Manufacturing Methods and Technology Measure For Arc Plasma Sprayed Phase Shifter Elements

1 July 1976 to 30 September 1976

Contract No. DAAB07-75-C-0043

Placed by

U. S. Army Electronics Command
Production Division
Production Integrated Branch
Fort Monmouth, NJ 07703

Raytheon Company
Research Division
Waltham, MA 02154

Distribution Statement

Approved for public release; distribution unlimited

NOTICES

Disclaimers

The findings in this report are not to be considered as an official Department of the Army position, unless so designated by other authorized documents.

The citation of trade names and names of manufacturers in this report is not to be construed as official Government indorsement or approval of commercial products or services referenced herein.

Disposition

Destroy this report when it is no longer needed. Do not return it to the originator.

Acknowledgement Statement

"This project has been accomplished as part of the U.S. Army (Manufacturing and Technology) (Advance Production Engineering) Program, which has as its objective the timely establishment of manufacturing processes, techniques or equipment to insure the efficient production of current or future defense programs."

Manufacturing Methods and Technology Measure

For Arc Plasma Sprayed Phase Shifter Elements

Fifth Quarterly Progress Report

1 July 1976 to 30 September 1976

Object of Study

"The objective of this manufacturing and methods technology measure is to establish the technology and capability to fabricate phase-shifter elements by the arc-plasma spraying techniques."

Contract No. DAAB07-75-C-0043

H. J. Van Hook
D. Massé
J. Saunders

Distribution Statement

Unclassified

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER 9
4. TITLE (and Subtitle) 6 Manufacturing Methods and Technology Measure for Arc Plasma Sprayed Phase Shifter Elements.		5. TYPE OF REPORT & PERIOD COVERED [redacted] Quarterly Progress rept. no. 5, 1 Jul [redacted] 30 Sep [redacted] 76,
		6. PERFORMING ORG. REPORT NUMBER 14 S-2112
7. AUTHOR(s) 10 H. J. Van Hook, D. Masse [redacted] J. Saunders		8. CONTRACT OR GRANT NUMBER(s) 15 DAAB07-75-C-0043
9. PERFORMING ORGANIZATION NAME AND ADDRESS Raytheon Research Division 28 Seyon Street Waltham, MA 02154		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 2759441
11. CONTROLLING OFFICE NAME AND ADDRESS U.S. Army Electronics Command Production Div., Production Integration Branch Fort Monmouth, NJ 07703		12. REPORT DATE 11 Oct [redacted] 76
		13. NUMBER OF PAGES 44 12 41 p.
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Arc plasma spraying	Phase-shifters
Ferrites	Microwave phase shifter materials
Dielectrics	Lithium ferrites

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Data on plasma spray parameters are given for 108 samples sprayed during this quarter. A new holding furnace and solenoid activated controls for sample translation are the major equipment changes that have been carried out this quarter. Several different ferrite powders and a number of dielectric compositions were investigated. Cracks in the ferrite layer, although reduced in number, are found in most samples. Typical values for coercive force of the annealed samples are 2.5 - 3.5 Oe; $4\pi M_r$ ranges from 500 to 800 gauss.

DD FORM 1 JAN 73 1473 EDITION OF 1 NOV 65 IS OBSOLETE

Unlimited

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

298320

ABSTRACT

Data on plasma spray parameters are given for 108 samples sprayed during this quarter. A new holding furnace and solenoid activated controls for sample translation are the major equipment changes that have been carried out this quarter. Several different ferrite powders and a number of dielectric compositions were investigated. Cracks in the ferrite layer, although reduced in number, are found in most samples. Typical values for coercive force of the annealed samples are 2.5 - 3.5 Oe: $4\pi M_r$ ranges from 500 to 800 gauss.

TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

LIST OF TABLES

GLOSSARY

Annealing - A heating schedule similar to firing but performed on a dense material to relieve strain, improve homogeneity or recrystallize a microcrystalline material.

Arc Plasma Spraying - High-temperature deposition technique in which molten or partially molten material is sprayed onto a heated substrate.

Coercive Force - The horizontal displacement of the magnetization vs applied field curve the hysteresis loop at zero induced field. A measure of the energy required to move magnetic domains through a solid material.

Core Material - The dielectric material which fills the hollow space within the ferrite toroid.

Dielectric - Oxide compounds which exibit polarization in electric fields.

Dilatometer - A device for measuring thermal expansion.

Elastic Modulus - The ratio of stress-to-strain (in pounds/in.^{2} or Newtons/in.^{2}) in isotropic materials which gives an indication of the stiffness or resistance to deformation. Also referred to as Young's modulus. Typically 10 to 50 $\times 10^6$ psi for oxides.

Ferrite - Oxide compounds of iron and other elements that exhibit a spontaneous magnetic moment due to magnetic spin dipole alignment within the structure.

Hysteresis Loop Properties - The display of magnetization vs applied field for a toroidal or long rod-shaped sample of a ferromagnetic material. The display, generally obtained or low frequencies (≤ 102 Hz) is useful in predictions of the magnetization properties and phase shift behavior at microwave frequencies ($\approx 10^{10}$ Hz).

Firing - Any high-temperature process performed on a material, but usually referring to a heating schedule which transforms a powder aggregate into a dense ceramic.

Isostatic Pressure - A powder compaction technique in which a sealed deformable container (e.g., a rubber bag with powder inside) is subject to a uniform compacting pressure from all sides.

Latched State - State of remnant magnetization after application of an applied field sufficient to magnetize in one or two opposite (180°) directions.

Lithium Ferrite - A class of ferrite materials with the general formula $Li_{.5+x/2-y2}Ti_xZn_{y2}Fe_{2.5-3x/2-y}O_4$ characterized by a saturation magnetization of $0 < 4\pi M_s < 3600$, a dielectric constant $18 < K < 20$, and frequently used in microwave devices.

Magnetic Compensation - A condition obtained in a specific ferrite composition and/or at specific temperatures where the magnetic moment is zero. At this point the opposed magnetic sublattices within the single phase composition exactly compensate.

Magnetometer - A device for measuring magnetic moment.

Microwave - That part of the electromagnetic spectrum between 100 MHz and 100 GHz.

Phase Shifter - A microwave device which serves as the active element in phased-array radar systems where the state of magnetic polarization is used to control the phase length of the electromagnetic energy. Also called phase control element.

Remanent Magnetization ($4\pi M_r$) - The value of induced field remaining in a material with toroidal geometry at zero applied field following the application of an applied field sufficient to uniformly magnetize a material.

Saturated Magnetization ($4\pi M_s$) - The saturation magnetization (c.g.s.) is the magnetic moment gauss/cm^3 of a material in an external DC field of sufficient magnitude to align the magnetic moment in the material parallel with it.

Saw Kerf - That portion of a solid removed by the cutting blade. The kerf width is usually about 5 percent wider than the width of the blade.

Spinel Ferrites - A class of iron oxide compositions having face-centered cubic crystal structures similar to the mineral spinel ($MgAl_2O_4$) and a magnetic moment which depends on composition.

Spray-Dried Powder - A form of powder aggregation where spherical particles of ~ 10 to 100 μm are produced which are themselves aggregates of much smaller (< 1 μm) particles. The advantage of this process is that the aggregates have better flow properties than untreated powder. The process is accomplished in a spray drier, a large funnel-shaped cavity into which a liquid suspension is sprayed and dried.

Stoichiometric - The idealized atomic proportions of elements in a chemical composition, such as the 1:2 in Mg:Al ratio in $MgAl_2O_4$. Departures from the exact integral proportions may have important effects on properties.

Stress-to-Failure - A statistical or average stress level of a solid where failure by brittle fracture propagation takes place, also called the modulus of rupture. Depends on surface conditions as well as intrinsic strength.

Thermal Expansion Coefficient - A parameter denoting the change in dimension ($\Delta l/l_o$) per unit temperature between ambient conditions and some elevated temperature. Since the actual expansion is not perfectly linear, one must specify the thermal interval of interest; i.e., $\alpha_{20^\circ}^{1000^\circ}$ = 15 ppm $^\circ C^{-1}$ denotes expansion between 20°C and 1000°C has our average slope $\Delta l/l_o \Delta T$ of $+15 \times 10^{-6}$ in./in./°C.

Toroid - A ring-shaped specimen used in magnetic measurements, particularly the hysteresis properties.

X-Ray Analysis - Analysis of crystal structure (X-ray diffraction), elemental composition (X-ray fluorescent analysis) to control processing or elucidate property variations using short wavelength radiation.

1.0 PURPOSE

The purpose of this program is to develop a manufacturing capability for producing the Patriot phase shifter element by arc-plasma spraying of a Li-Ti-ferrite onto a dielectric substrate. The primary objective is to produce the phase control element as a finished composition with acceptable microwave properties and a reasonably high yield. To achieve sound composites, one of the properties needing constant monitoring is the match in thermal expansion coefficient between the ferrite coating and the dielectric. A second important area for control and reproducibility is the thermal environment during spraying. Thermal conditions are influenced mainly by arc current, the gas velocities, and the substrate-to-gun separation distance. Finally, to achieve a low unit cost, it is necessary to improve yield and reduce machining costs by working with local machine shops to improve overall efficiency.

2.0 NARRATIVE AND DATA

2.1 Preparation and Testing of Starting Materials

As we solve the problems of equipment functioning and begin to produce larger quantities of plasma-sprayed phase shifters, the improtance of control and reproducibility in the starting materials becomes increasingly evident. In this last quarter one hundred and eight APS samples were sprayed. One of the serious problems we encountered was the cracking of the thin dielectric parts during transfer and spray operations. This problem has been reduced to tolerable levels (fewer than 10 percent failures) by improvements in operator technique and equipment stability, by changing the phase-shifter dielectric cross section and by altering the firing conditions. The solution to processing and manufacturing problems of this sort must consider each step of the operation (from initial powder processing to final arc-plasma spray technique) to minimize each of the contributing factors.

The flow uniformity of the ferrite powder is one important factor affecting the reproducibility of the APS process. The powder flow must be unimpeded by agglomeration or moisture, and the hopper feed must function smoothly to avoid spitting or fluctuating delivery as well as uneven buildup on the target. During this quarter we had difficulties with powder delivery. Again the problem was solved by altering several process steps: (1) the powder was dried just before plasma spraying; (2) the powder was screened for greater uniformity; and (3) the powder distribution wheel in the feed hopper was replaced. The distribution wheel, a 3-inch diameter disk-shaped part, was only .002 in. out of flatness, but this was sufficient to cause a noticeable pulsation in the feed.

2.1.1 Dielectrics

During this quarter a number of bars were pressed and fired from dielectric powders on hand. A new composition which has been used extensively is type LMTF 195, which has a thermal expansion coefficient slightly larger than the standard LMTF 190. The increased Li-Ti substitution of LMTF 195 reduces $4\pi M_s$ to negligible values, which is an advantage over the LMTF 190 composition where $4\pi M_s = 90$ gauss (assuming the magnetization here should be zero).

The expansion coefficient α increases steadily with temperature, i.e., the expansion curve when plotted as $\Delta \ell / \ell_0$ (ordinate) and temperature (abscissa) has a continuous upward curvature; therefore one must choose a specific temperature to compare values. We have chosen 1000°C as the critical temperature, corresponding to T-A = 1000° - 20° = 980°C on the expansion curves in previous reports (A = ambient temperature). The values now used for α at 1000°C are shown in the following table.

TABLE I

THERMAL EXPANSION COEFFICIENT AT 1000°C FOR VARIOUS SPINEL DIELECTRICS

		α values in ppm/°C at 1000°C		
Designation	x*	w* = 0	w = .10	w = .15
LMTF 200	1.00	15.4	15.1	15.0
LMTF 195	.975	15.25	15.0	14.85
LMTF 190	.95	15.1	14.9	14.7
LMTF 180	.90	14.9	14.7	

* $Li_{.5+x/2}Mn_{.1}Ti_xAl_wFe_{2.4-3x/2-w}O_4$

The value of α is an average value $\alpha = \Delta \ell / \ell_0 (T-A)$ taken from data taken from a continuous curve run to 980°C and extrapolated to 1000°C.

As we will show in the discussion on individual runs (Sec. 2. 2. 2) and the hysteresis results (Sec. 2. 2) there is no clear correlation between cracking and hysteresis properties on one hand and dielectric composition on the other. This means that other factors in the APS process still dominate the final properties. However, we expect the expansion properties to enter in the final stages of perfecting the APS manufacturing process.

2. 1. 2 Ferrite powder evaluation

The ferrite powders used for APS deposition during this quarter were from two 40 kgm Raytheon SMDO batches, LMTF 50 (G3) and LMTF 50 (G4). These powders were carefully characterized by X-ray diffraction, X-ray fluorescence, scanning electron microscopy and surface area analysis. Table II gives X-ray and particle size (diameter) data on the three batches used to date. We include results on the LMTF 53 (G2) powder used in the original APS experiments at ECOM laboratories in July 1975. This powder has a higher Li-Ti content, since in the original formulation $x = 0.53$, compared with $x = 0.50$ for G3 and G4 powders. As reported previously, this G2 powder fired conventionally to full density gave $4\pi M_s$ = 1150 gauss. Similar firings of the G3 ferrite have given $4\pi M_s$ = 1250 gauss.

TABLE II

PHYSICAL PROPERTIES OF SPRAY-DRIED Li-Ti-FERRITE USED IN PLASMA SPRAY EXPERIMENTS

Designation	Lattice par. (A)	Spray dried avg. part. diam. (μm)	BET surface area (m^2/gm	Equiv. part. size (μm)
LMTF 53 (G2)	8.345	5.5		
LMTF 50 (G3)	8.346	4.7 f* 7.5 ch*	2.2	0.87
LMTF 50 (G4)	8.346		4.3	0.45

* f = fines fraction, * ch = chambers fraction.

The tabulated data show that there is no significant difference in lattice parameter which would indicate variation in iron content. There was no separation of spray-dried fractions in the G2 powder, but the size fraction (Fig 7, 2nd Qtrly. Report) is not significantly different from the averaged histograms for the G3 powder (Fig. 7, 3rd Qtrly report).

The surface area measurement and the calculated average particle size refer to the size of the individual particles which make up the "eggshell" geometry (Fig. 1) of the spray-dried particle agglomerate. A larger surface area (smaller particle size) indicates a greater efficiency in the final milling of the calcined powder before spray drying. More milling action and particle size reduction should yield a more homogeneous powder, and one

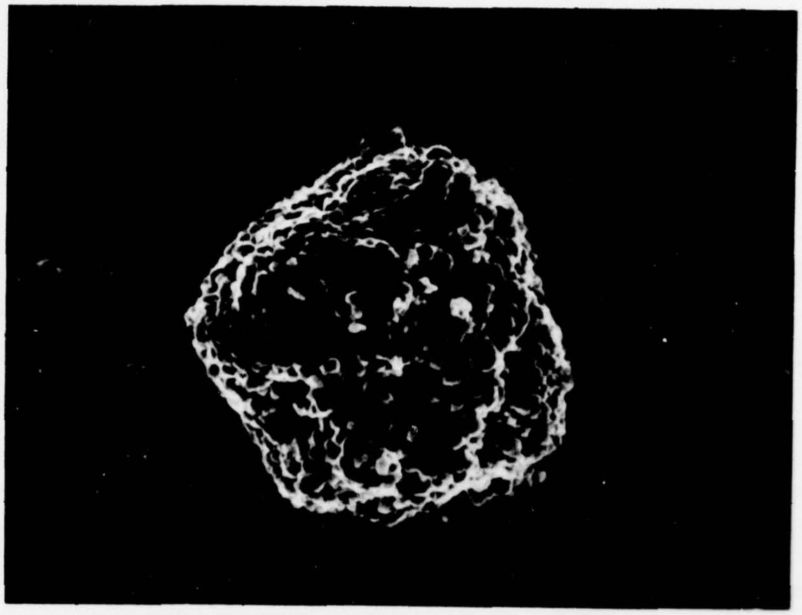


Figure 1. Photograph at 2000× of a Typical Spray-Dried Particle.

which melts more quickly in the plasma flame. The differences in surface between G3 and G4 powders is surprisingly large considering the similar processing (two calcines, three millings) these powders were given. The measurements must be rechecked for accuracy.

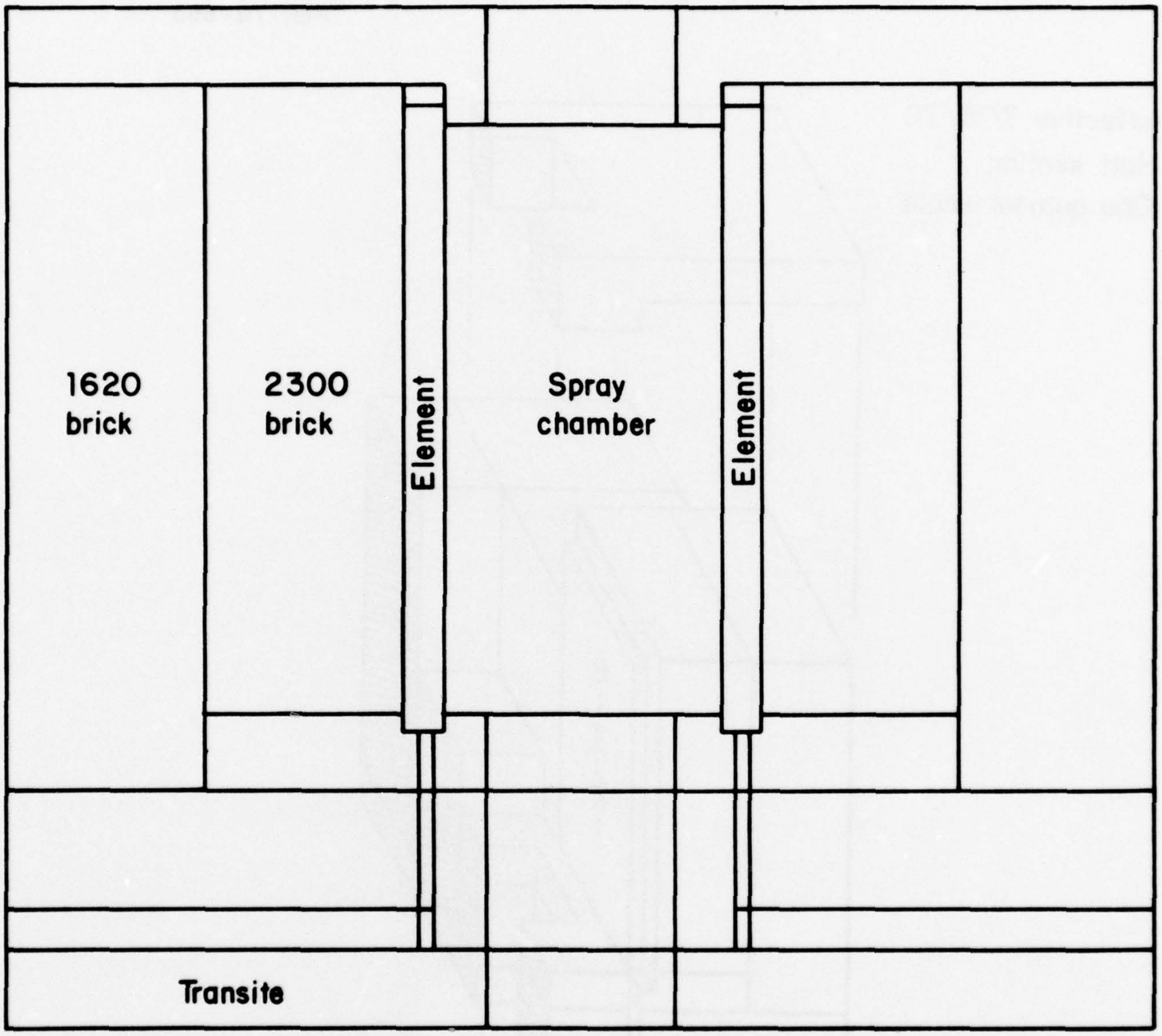
2.2 APS Experiments at Raytheon

2.2.1 Equipment modifications

Plasma spray runs No. 110 and following made use of the redesigned and rebuilt furnace shown in Figs. 2 and 3. We had experienced two problems with the cylindrical spray chamber: (1) the chamber temperature reached only 600°C during spraying, although 700° - 750°C was desired: and, (2) an excessive number of ferrite overspray particles floated about the chamber during spraying. The second problem was related to the first, in that larger exhaust ports could be used to remove the spent powder that missed the substrate, although larger ports would mean more draft and increased difficulty in maintaining temperature. The present furnace is a rectangular box ($8 \times 7 \times 3.5$ in.) with two 4×8 in. kanthal heating elements on the side walls. The furnace volume is larger by a factor of two than the earlier cylindrical oven but the power seems adequate ($\approx$ 1800 watts) to maintain temperature during spraying.

The holding oven above the spray chamber is generally maintained at 600°C during spraying and transfer steps. Although there is danger of cracking (thermal shock) the uncoated substrates, we feel this risk is more acceptable than the thermal shock of bringing finished samples from the 750°C spray chamber up into a much lower-temperature holding oven.

Twenty-four samples were sprayed in the second half of July after work on the oven rebuilding was completed. The APS numbers were 110 to 133 inclusive. In these runs we used several new anode configurations to improve deposition. One of these variations was to change the angle of entrance of the powder feed from near 90° to a forward angle of 54°, bringing the powder out 0.250 in. further downstream than the standard 901-11



Effective 7/15/76

One half scale

Figure 2 Spray Chamber Furnace (One-half scale).

PBN-76-663

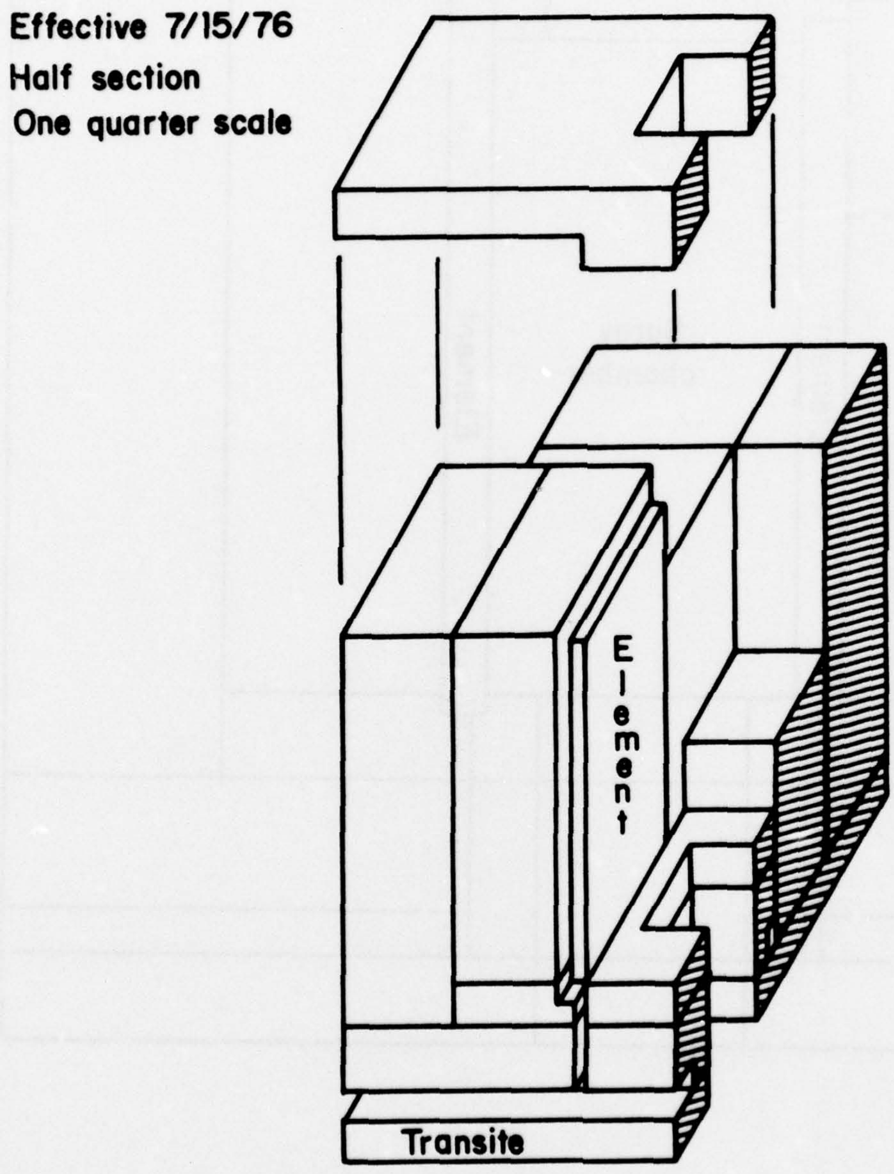


Figure 3 Spray Chamber Furnace (One-quarter Scale).

shape. The result was unsatisfactory in that very high arc currents were needed to heat the powder enough to adhere to the substrate. In recent runs we have gone back to the standard 901-12 anode (ID = .187 in.) with perpendicular powder feed and have moved the gun closer (to 3.5 in. and 3.25 in.), with much better results.

The hydraulic mechanism for vertical motion of the APS tube was modified this quarter. Solenoid values were installed, in parallel with the slow speed needle valve controls, to allow rapid motion in the up or down direction. The rapid translation is controlled by a three-way toggle switch which opens one of the normally closed solenoid valves, bypassing the needle valve on that side of the line. In practice we find that this arrangement moves the sample too rapidly from spray furnace to holding furnace and a manual override of the automatic controls is generally used to slow down the motion. As the process becomes standardized we will probably make more use of the solenoid valves.

2.2.2 Description of individual runs and hysteresis properties on machined samples

The APS runs performed this quarter are summarized in the spray log (Table III). In this section we include any data on hysteresis properties because this measurement is a good indicator of microwave phase shift performance. It is important to correlate the magnetic properties with APS conditions to control the manufacturing process.

APS run 110 was the first which used the rebuilt spray furnace with the enlarged rectangular spray chamber. This run was largely experimental with the spray chamber at 800°C, helium added to the argon arc gas, and higher arc current values. The powder flow of the LMTF 50 (G4) fines was poor, probably because of moisture or lack of screening.

Run 111 was also experimental. Helium gas was not added, and lower arc current was used. The depth of the spray chamber was increased

TABLE III

ARC PLASMA SPRAY LOG
High Velocity Nozzle

Date	Number	Ferrite	Dielectric	Current Amps	Gas Flow - CFH Arc	Gas Flow - CFH Powder	Hopper Speed	Spray Distance in	Rate Rot %	Rate Pull in/min	Furnace Chamber	Temperature Holding	Anneal Cycle*	Comments
7/15/76	110	LMTF50G-4 Chambers	LMTF195(12)	220-600	Ar 40 He 7 1/2	O_2 15	50-70	4	50	.8	800°	600°	1010°-1 1/2 Hrs. (H)	New furnace - Works great - Chamber should be deeper - Anode worked poorly - Powder flowed poorly
7/16/76	111	LMTF50G-4 fines	LMTF190(36)	320-340	Ar 40	O_2 15-13	60-75	4	50	.6 - .55	725°	600°	1010°-1 1/2 Hrs. (H)	Chamber deepened -better
	112			380	Ar 40	O_2 13-7 1/2	75	4	50	.8	750	600		
	113			400-500	Ar 50-45	O_2 15	70	4	50	.7	750	600		
	114			420	Ar 40	O_2 13	80	4	50	.8	750	600		
	115			380	Ar 35	O_2 15	65	4	50	.92	750	600		Changed from large anode to 901-12 anode (regular)
	116			Substrate cracked - aborted										
	117			400	Ar 35	O_2 15	70	4	50	.85	750	600		Big red glow - But best depositing conditions - even buildup negligible
7/21	118	LMTF50G-4 Fines	LMTAF2007A	400	Ar 40	O_2 15	70	4	50	.77	750	600	1010°-1 1/2 Hr. (H)	Powder flow a problem

* (H) refers to APS holding oven. II and III refer to separate Lindberg furnaces for annealing.

ARC PLASMA SPRAY LOG (Cont'd.)

High Velocity Nozzle

Date	Number	Ferrite	Dielectric	Current Amps	Gas Flow - CFH Arc	Gas Flow - CFH Powder	Hopper Speed %	Spray Distance in	Rate Rot %	Rate Pull in/min	Furnace Temperature Chamber	Furnace Temperature Holding	Anneal Cycle*	Comments
7/21/76	119	LMTF50G-4	LMTAF180(33)	400	Ar 40	O_2 15	60	4	50	.8	750	600	1010^0-1 1/2Hr(H)	Powder flow a problem
7/22	120	LMTF50G-4	LMTAF180(33)	400	Ar 40	O_2 15-19	60-70	4	50	.65-.8	750	600	1010^0-1 1/2 Hr(H)	Powder freshly dried overnight @ 80^0C
	121			400	Ar 40	O_2 20	70	4	50	.83	750	600		Sample broke halfway
	122			300	Ar 40	O_2 17	70	4	50	.85	750	600		Reduced current still producing red glow - tho not as bright
	123			300	Ar 40	O_2 18	70	4	50	.85	750	600		
7/26/76	124	LMTF50G-4	LMTAF180(33)	300	Ar 40	O_2 17	75	4	50	.85	750	600	1010^0-1 1/2 Hr(H)	
	125			300	Ar 40	O_2 17	75	3 1/2	50	1.0	750	600		D_s decrease improved deposit after powder port
	126	LMTF50G-4 Fines	LMTAF180(33)	240	Ar 40*	O_2 17	75	3 1/2	50-40	0.95	750	600		Holding furnace TC still not near samples
	**127			240-500	Ar 40	O_2 17	60	3 1/2	50	0.85	750	600	901-10 Anode did not work with usual parameters	
7/28	128	LMTF50G-4 Fines	LMTF190(36)	200-220	Ar 40*	O_2 17	70	3 1/2	50	0.72	750	700	1010^0-1 1/2Hrs(H)-O_2	Current too low
	129			220-240	Ar 40*	O_2 17	70	3 1/2	40-50	.70-.82	750	700	Temp. actually went to 1060 for 10 min.	
	130			230-240	Ar 40	O_2 17	70	3 1/2	50	.87	750	700		7 Min spray plus 3 min transfer - 10 mins. total

* (H) refers to APS holding oven. II and III refer to separate Lindberg furnaces for annealing.

ARC PLASMA SPRAY LOG (Cont'd.)
High Velocity Nozzle

Date	Number	Ferrite	Dielectric	Current Amps	Gas Flow - CFH Arc	Gas Flow - CFH Powder	Hopper Speed %	Spray Distance in	Rate Rot %	Rate Pull in/min	Furnace Temperature Chamber	Furnace Temperature Holding	Anneal Cycle*	Comments
7/28	131	LMTF50G-4 Fines	LMTF190(36)	250	Ar 40	O_2 17	70	3 1/2	50	.92	750	700		Approx. 34 grams deposited
	132			250	Ar 40	O_2 17	70	3 1/2	50	.95	750	700		
	133			250-840	Ar 50-45	O_2 17	70	3 1/2	50	.95	750	700		Arc gas flow of 50 CFH - Too high
8/3	134	LMTF50G-4 Fines	LMTF190(36)	240	Ar 40	O_2 17	70	3 1/2	50	.80	750	700	1010°- 1 1/2 Hr(H) TC moved near samples - Temp actually 1050 - 15 min 1010 - 1 hour	Approx. 121 grams G-4 powder per sample in this run
	135			240-260	Ar 40*	O_2 17	70	3 1/2	50	.95	720	700		A Quick Anneal 135, 138 1 800° - 40 min 2 1000° - 1 hour
	136			260	Ar 40*	O_2 17	70	3 1/2	50	.98	750	700		
	137			260	Ar 40	O_2 17	70	3 1/2	50	1.0	750	700		
	138			255	Ar 40*	O_2 17	70	3 1/2	50	1.0	740	700		
	139			260	Ar 40	O_2 17-15	70	3 1/2	50	.9	740	750		
8/4	140	LMTF50G-4 Fines	LMTF200(1)	260	Ar 40	O_2 17	60	3 1/2	50	.75	750	700	1010°-1 1/2Hrs. TC located through front brick	
	141			260	Ar 40	O_2 17	60	3 1/2	50	.75	750	700		
	142			260	Ar 40	O_2 17	60	3 1/2	50	.85	750	700		
	143			260	Ar 40	O_2 17	60	3 1/2	50	.95	750	700		

* (H) refers to APS holding oven. II and III refer to separate Lindberg furnaces for annealing.

ARC PLASMA SPRAY LOG (Cont'd.)

High Velocity Nozzle

Date	Number	Ferrite	Dielectric	Current Amps	Gas Flow - CFH Arc	Gas Flow - CFH Powder	Hopper Speed %	Spray Distance in	Rot %	Rate Pull in/min	Furnace Temperature Chamber	Furnace Temperature Holding	Anneal Cycle*	Comments
8/4	144	LMTF50G-4 Fines	LMTF200(1)	260-280	Ar 40	O_2 17	60	3 1/2	50	.85	750	700	1010^{o}-1 1/2Hrs.	
	145		LMTF195(12)	280	Ar 37 1/2	O_2 17	60	3 1/4	50	1.1	750	700		
	146			280	Ar 37 1/2	O_2 17	60	3 1/4	50	1.5	750	700		
8/26	147	LMTF50G-4 Fines	LMTAF200(2)	280	Ar 37 1/2	O_2 17	60	3 1/4	50	1.0	700	700	No Anneal	Holding Furnace TC located on furnace floor with bead bent into furnace area - Temperature too high - Samples broke because of thermal shock
	148			270	Ar 37 1/2	O_2 17	60	3 1/4	50	1.0	700	650		
	149			270	Ar 37 1/2	O_2 17	60	3 1/4	50	1.0	700	650		
	150			280	Ar 37 1/2	O_2 18	60	3 1/4	50	1.0	700	650		
	151			280	Ar 37 1/2	O_2 18	60	3 1/4	50-40	1-.95	700	650		
	152			280	Ar 37 1/2	O_2 18	60	3 1/4	50	1.0	700	650		
	153			280	Ar 37 1/2	O_2 18 1/2	60	3 1/4	50	0.95	700	875-900	No Anneal	Holding Furnace TC malfunctioned - Temp too high
	154			280	Ar 37 1/2	O_2 18 1/2	60	3 1/4	50	0.95	700	875-900		
	155			280	Ar 37 1/2	O_2 18 1/2	60	3 1/4	50	.95	700	875-900		First sample sprayed from top down
8/31	156	LMTF50G-4 Fines	LMTF190(36)(1/2) LMTAF190-15A(1/2)	290	Ar 36	O_2 18 1/2	60	3 1/4	50	.92	700	650		New chamber elements - New powder dist. wheel
	157	Dried 4 hrs. @ $100^{o}C$	LMTAF200-7A	290	Ar 36	O_2 17	65	3 1/4	50	.92	700	650		
	158			290	Ar 36	O_2 17-15	60	3 1/4	50	.92-.8	700	650	1015^{o}-1 1/2Hr Air	Only bottom to top spray in run - Only sample to crack in anneal

* (H) refers to APS holding oven. II and III refer to separate Lindberg furnaces for annealing.

ARC PLASMA SPRAY LOG (Cont'd.)
High Velocity Nozzle

Date	Number	Ferrite	Dielectric	Current Amps	Gas Flow - CFH Arc	Gas Flow - CFH Powder	Hopper Speed %	Spray Distance in	Rate Rot %	Rate Pull in/min	Furnace Temperature Chamber	Furnace Temperature Holding	Anneal Cycle*	Comments
8/31	159	LMTF50G-4	LMTAF200-7A	290	Ar 36	O_2 17 1/2	65	3 1/4	50	1.0	700	650	1015^{o}-1 1/2Hr Air (II)	
	160			290	Ar 36	O_2 17 1/2	65	3 1/4	50	1.0	700	650		
	161			290	Ar 36	O_2 17 1/2	65	3 1/4	50	1.0	700	650		10 samples sprayed in 2 hrs. - approx. 1025 grams All "downers"
	162			290	Ar 36	O_2 17 1/2	65	3 1/4	50	1.0	700	650		
	163			290	Ar 36	O_2 17 1/2	65	3 1/4	50	1.0	700	650	1015^{o} III-1 1/2Hr-O_2	
	164			290	Ar 36	O_2 17 1/2	65	3 1/4	50	1.0	700	650		
	165			290	Ar 36	O_2 17 1/2	65	3 1/4	50	0.95	700	650		
9/1	166	LMTF50G-4 Fines	LMTF200(2)	200	Ar 36	O_2 17 1/2	65-70	3 1/4	50	0.92	700	650	1015^{o}(II)-1 1/2Hrs-O_2	
	167			280	Ar 36	O_2 17 1/2	70	3 1/4	50	.95	685	650		
	168		LMTF200(1)	280-260	Ar 36	O_2 17 1/2	70	3 1/4	50	.95-.85	700	650		
	169			310	Ar 36	O_2 17 1/2	65-70-80	3 1/4	50	1.2	700	650		
	170			305	Ar 36	O_2 17 1/2	75	3 1/4	50	1.3	700	650		
	171			305	Ar 36	O_2 17 1/2	75-70	3 1/4	50	1.3	700	660		Slight gun buildup at powder feed of 75
	172		LMTAF180(33)	305	Ar 36	O_2 17 1/2	70	3 1/4	50	1.3	700	665		
	173			305	Ar 36	O_2 17 1/2	70	3 1/4	50	1.3	700	665		Top inch blown off but spray completed
	174		LMTAF200-7A	305	Ar 36	O_2 17 1/2	70	3 1/4	45-50	1.3	700	650		Rotation erratic - slower speed
	175			305	Ar 36	O_2 17 1/2	70	3 1/4	50	1.3	700	650		

* (H) refers to APS holding oven. II and III refer to separate Lindberg furnaces for annealing.

ARC PLASMA SPRAY LOG (Cont'd.)
High Velocity Nozzle

Date	Number	Ferrite	Dielectric	Current Amps	Gas Flow - CFH Arc	Gas Flow - CFH Powder	Hopper Speed %	Spray Distance in	Rate Rot %	Rate Pull in/min	Furnace Temperature Chamber	Furnace Temperature Holding	Anneal Cycle*	Comments
9/2/76	176	LMTF50G-4 Fines	LMTAF180(33)	300	Ar 36	O_2 17 1/2	70	3 1/4	50	1.1	700	650	1015^0(III)-1 1/2Hrs-O_2	First sample after trying undried G-3 powder
	177		LMTAF190-15A	300	Ar 36	O_2 17 1/2-18 1/2	70	3 1/4						
	178			300	Ar 36	O_2 18 1/2-16	70	3 1/4	50	1.0	700	650		
	179		LMTAF180(33)	315	Ar 36	O_2 17	70	3 1/4	50	1.0	710	670		
	180		LMTAF190-15A	320	Ar 36	O_2 17	70	3 1/4	50	1.3	710	665		
	181			320	Ar 36	O_2 17	70	3 1/4	50	1.3	700	665		
	182			310	Ar 36	O_2 17	70	3 1/4	50	1.3	700	665		
	183			300-320	Ar 36	O_2 17	70	3 1/4	50	1.3	700	665		Better deposit at 320 amps
	184			320	Ar 36	O_2 17	90	3 1/4	50	1.4	700	665		Fastest deposit to date
9/14	185	LMTF50G-3 Fines	LMTAF200(2)	320	Ar 36	O_2 16	65	3 1/4	50	1.0	700	650	1025^0(II)-1 1/2Hrs-O_2	No a smooth spray - first sample roughness
	186		LMTAF190-15A	330	Ar 36	O_2 16 1/2	65	3 1/4	50	.7-1.0	700	650		Very wobbly
	187		LMTF195(11)	330-320	Ar 36	O_2 16 1/2	70	3 1/4	50	1.3	700	650		First two substrates broke during spraying
	188			360	Ar 36	O_2 16 1/2	70	3 1/4	50	1.3	700	650		Current crept up
	189			310	Ar 36	O_2 17 1/2	65	3 1/4	50	1.0	700	650	1015^0(III)-1 1/2Hrs-O_2	Continuous trouble up to this point with falling stringers that are 1/2 in. long then fall off
	190			310	Ar 36	O_2 17	65	3 1/4	50	1.1	700	665		
	191			310-340	Ar 36	O_2 18	65	3 1/4	50	.95	720	650		Current surging during run

* (H) refers to APS holding oven. II and III refer to separate Lindberg furnaces for annealing

ARC PLASMA SPRAY LOG (Cont'd.)
High Velocity Nozzle

Date	Number	Ferrite	Dielectric	Current Amps	Gas Flow - CFH Arc	Gas Flow - CFH Powder	Hopper Speed %	Spray Distance in	Rot %	Rate Pull in/min	Furnace Temperature Chamber	Furnace Temperature Holding	Anneal Cycle	Comments
9/14	192	LMTF50G-3	LMTF195(11)	320	Ar 37	O_2 18	65	3 1/4	50	1.0	700	650	1015°-1 1/2 Hr-O_2	Arc gas increased to check surging - Sample broke at base - Left in chamber
	193			320	Ar 37	O_2 18	65	3 1/4	50	1.0	700	650		
	194			Sample broke										
9/15	195	LMTF50G-4 Chambers	LMTF195(12)	320	Ar 37	O_2 17-10	65	3 1/4	50	0.85	700	650		Powder gas decrease stopped stuttering feed
	196	-170 Mesh (-88μ)		360	Ar 37	O_2 13	65	3 1/4	50	1.0	700	650		Excellent parameters
	197			360	Ar 37	O_2 13	65	3 1/4	50	1.0	700	660		
	198			340	Ar 37	O_2 13	70	3 1/4	50	1.2	700	665		
	199			350	Ar 37	O_2 13	65	3 1/4	50	0.95	700	665		Low arc gas tand volume influenced current fluctuation
	200			340	Ar 38	O_2 13	65	3 1/4	50	1.0	700	665		Powder ran out - Overlapped in middle
9/21	201	LMTF50G-4 Chambers -88μ	LMTF195(12)	340	Ar 37	O_2 13	65	3 1/4	50	1.2	700	650	1200°-30 min-O_2	
	202			340-400	Ar 37	O_2 11	65-75	3 1/4	50	1.2	700	650		Current surged
	203			400	Ar 37	O_2 11 1/2	75	3 1/4	50	1.2	700	650		
	204			400-460	Ar 37	O_2 11	65	3 1/4	50	1.1	700	650		Current crept up
	205			440-460	Ar 37	O_2 11	65	3 1/4	50	1.3	700	650		Excellent deposit
	206			460-500-440	Ar 37	O_2 11	65	3 1/4	50	1.3	700	650		Current above 460 appeared to melt powder
	207			440	Ar 37	O_2 11	65-75	3 1/4	50	1.3-1.6	700	650	1200°-30 min-O_2 Controller malfunctioned	
	208			480	Ar 50	O_2 17-13	65	3 1/4	45	0.7				Attempt to similate APS 12 with higher velocity

ARC PLASMA SPRAY LOG (Cont'd.)
High Velocity Nozzle

Date	Number	Ferrite	Dielectric	Current Amps	Gas Flow - CFH Arc	Gas Flow - CFH Powder	Hopper Speed %	Spray Distance in	Rot %	Rate Pull in/min	Furnace Temperature Chamber	Furnace Temperature Holding	Anneal Cycle*	Comments
9/21/76	209	LMTF50G-4 -88μ	LMTF195(12)	480	Ar 50	O_2 13	65-55	3 1/4	45	0.8	700	650	1200^0(H)-30 min-O_2 Controller malfunctioned	Too much waste overspray at hopper 65
	210	↓	↓	440	Ar 38	O_2 13	65	3 1/4	45	1.3	700	650	↓	
9/23	211	LMTF50G-4 -88μ	LMTAF190-15A	360	Ar 38	O_2 13	65	3 1/4	50	1.1	700	650	1020^0(H)-2Hrs-O_2	New TC in holding furnace - Completely sleeved - located close to ceiling over floor hole - APS 212 appeared warped after spraying
	212		LMTAF195-10A	420	Ar 38	O_2 13	65	3 1/4	50	1.2	700	650		
	213			400	Ar 38	O_2 13	65	3 1/4	50	1.2	700	650		
	214			400	Ar 38	O_2 13	65	3 1/4	50	1.0	700	650		Looks warped
	215			400	Ar 45	O_2 13	65	3 1/4	50	0.9	700	650		
	216			400	Ar 45	O_2 13	65	3 1/4	50	0.95	700	650		
	217	↓	↓	400	Ar 45	O_2 13	65	3 1/4	50	1.0	700	650	↓	

* (H) refers to APS holding oven. II and III refer to separate Lindberg furnaces for annealing.

by moving the rear bricks back, which reduced the powder turbulence (because of the enlarged volume).

APS run 111 used a larger diameter anode (Part No. 901-11) which the manufacturer designated suitable for spraying refractory ceramics. Our purpose in investigating the larger-bore anode (0.250 in. vs 0.187 for the standard high-velocity anode) was to reduce overheating of the dielectric substrate and minimize cracking in the ferrite layer near the interface by spreading the plasma flame. The use of this anode in runs 111 through 114 reduced deposition rate and led to very high powder losses. Samples 112, 114 and 115 were machined to dimension and annealed at 1010°C for 1.5 hours. Measurement of H_c and B_r after a 15 ampere latching pulse are shown in Table IV. The samples received a second 800°C anneal for 2 hours.

TABLE IV

HYSTERESIS PROPERTIES OF APS SAMPLES 112, 114 AND 115

		1010°C anneal		1010° and 800°C anneal	
No.	Length (in.)	H_c	B_r	H_c	B_r
112	1.100	2.89	508	2.31	574
114	1.505	3.66	679	2.66	705
115	1.575	3.76	682	2.69	475

Sample 112 shows a rather low B_r (508 gauss) which was improved slightly by the second anneal. Sample 114, sprayed at a higher arc current, shows much better B_r. Sample 115, sprayed as was 114 except that the standard bore (0.187 in.) high-velocity anode and lower arc current were used, showed H_c and B_r similar to 114 after the first anneal cycle. In the second anneal B_r on 115 was abruptly reduced, evidently because of new cracks in the ferrite layer.

Runs were made on July 21 and 22 using different dielectric substrates and standard current, gas flow and hopper feed conditions to see if physical properties correlated with substrate material. In these runs we experienced difficulty with clogging and uneven delivery of the G4 powder, which gave very irregular as-deposited shapes. Only one sample in this series was machined to cross section and measured (No. 120); this gave $H_c = 2.32$ $B_r = 657$ after the standard 1010°C anneal.

During this time we also evaluated other modifications to the high-velocity anode. Run No. 127 used the 0.250 in. ID anode with the powder feed port angled forward to 55° rather than the usual near-90° angle between arc gas channel and powder feed port. We also began to experiment with a closer spray distance (3.5 rather than the 4 in. used earlier).

The spray run on July 28 was an attempt to produce a number of samples under nearly identical conditions. The dielectric was the 190 composition used extensively at ECOM Laboratories, the powder was the (G4) fines, spray distance was maintained at 3.5 in., the arc current kept low at 200 - 240 amperes. Pull rates for this series were near 1 in./min., largely because of the closer spray distance which improved the capture cross section of the spray. With a spray rate of 10 minutes per full-length sample and ≈ 34 grams deposited per sample, this represents the best process efficiency we have achieved with the APS process, before or since this date. The overspray of ferrite on those samples was about 15 gm for the ≈ 6 in. sprayed length. The total powder used per sample was ≈ 125 gm, indicating a deposition efficiency of 34/125 = 0.27 and a ratio of total ferrite powder weight of the 0.050 in. ferrite layer of 19/125 = 0.15. It is unlikely that this degree of efficiency can be improved without moving the gun substantially closer to the dielectric.

The anneal following the spray run went substantially higher than planned (to 1060°C). After machining, these samples showed more cracks than usual, which we ascribe to the excessive temperature.

The APS run on August 4 produced samples 140 through 146, all of which were full-sized elements. After the standard 1010°C anneal the following loop properties were obtained.

TABLE V

HYSTERESIS PROPERTIES OF APS 141 THROUGH APS 146 AFTER ANNEALING

Designation	Length	H_c	B_r
APS 141	5.142	2.78	673
APS 142	5.145	2.79	641
APS 143	5.145	2.74	626
APS 144	5.145	2.79	661
APS 145	5.145	3.00	566
APS 146	5.145	2.40	587

Although H_c is acceptably low in these samples, B_r is too low by 50-75 gauss to give the required 340° phase shift.

The APS run on August 26 was another attempt to maintain constant spray conditions which would provide reproducible samples for the confirmatory materials delivery. Arc current was held at 280 amps, the arc gas flow at 37.5 CFM, and pull rate of 1 in./minute. Nine good samples were produced in this run. Unfortunately, in the heat treatment immediately following the run, the holding furnace went well above the planned temperature and all of these samples were lost. The furnace control thermocouple had been relocated to avoid controller problems due to ac pickup from furnace windings. Instead, the new thermocouple location intensified the pickup, leading to controller malfunction and failure of the anneal.

Microscopic examination of a number of APS samples indicated a very low ferrite density at the dielectric interface grading rapidly into the normal ferrite coating density. We hypothesized that this low density deposit was caused by relatively cool ferrite from the periphery of the spray pattern. To eliminate the deposition of this material preceding the hotter deposits from the center of the plasma, we experimented with metal shields to mask the uncoated dielectric rod. However, with material accumulating on the shield and the shield obscuring a view of the sample, these experiments proved unsuccessful.

By reviewing the earlier work at ECOM, and by consulting with R. Babbitt, we found another solution: to spray the top (free) end of the sample first, moving downward to the attached end (rather than the reverse, as we had been doing). The disadvantage of this approach is that the torque applied by plasma spray pressure and the moment generated by the weight of the coating on the free end can snap the dielectric rod. The advantage is that the overspray powder is convected upward and does not deposit prematurely on the substrate.

In the next APS series (August 28) the samples were sprayed from the top (free) end downward to the base attachment. Since the technique gave significant improvement in density at the interface, we have continued this method in subsequent runs. Runs APS 156 through APS 165 were again sprayed under standardized conditions: type LMTF 200 (7A) substrate, constant spray-chamber temperature (700°C), and holding temperature oven (650°C).

The hysteresis properties after anneals at 1015°C and 800°C are shown in Table VI.

TABLE VI

HYSTERESIS LOOP PROPERTIES OF APS SAMPLES PRODUCED ON8-31-76

Designation	Length (in.)	H_c (Oe)	$4\pi M_r$ (gauss)
APS 159	5.145	3.20	593
APS 160	5.145	3.37	635
APS 161	5.145	3.37	728
APS 162	5.145	3.19	731
APS 163	5.145	3.22	705
APS 164	5.145	2.75	529
APS 165	4.705	3.31	780

Three of the above samples with $4\pi M_r > 725$ gauss would have a phase shift per unit length larger than the accepted minimum. Unfortunately, sample APS 165 was thin on one end and could not be made to the acceptable length. If the sample had been full length, its phase shift would have been 362°.

The most problematic data shown in Table VI are the samples with unexplainably low $4\pi M_r$ such as APS 159 and APS 164. These were sprayed identically with those showing higher $4\pi M_r$ onto the same substrates and annealed together for strain relief and recrystallization. One can always speculate that cracks in samples 159 and 164 reduced $4\pi M_r$: however, our external examination of samples 161 and 162 revealed at least as much visible cracking, although $4\pi M_r$ was ~ 30 percent higher. Some very recent studies on dissected phase shifters (Sec. 2.3) indicate that this anomaly is probably caused by nonuniform ferrite walls in distorted samples. The wall non-uniformity can be seen only by destructive sectioning of the elements.

The next plasma run (September 1) again used the type LMTF 50 (G4) fines fraction, spray-dried powder and the top-down method of deposition to avoid overspray onto the bare dielectric. The different dielectrics used were the LMTF 200 ($\bar{\alpha}$ = 15.4 ppm/°C), the 180 (33) material (α = 14.7 ppm/°C), and the 200 (7A) composition (α = 15.2 ppm/°C). These were sprayed under nearly identical conditions. Arc current and hopper feed were set slightly higher, allowing somewhat faster deposition rates (1.3 in./min or ≈ 5 minutes per 6.5 in. sprayed length). The hysteresis loop properties on machined and annealed samples in this series are shown in Table VII.

TABLE VII

HYSTERESIS LOOP PROPERTIES OF APS SAMPLES PRODUCED ON 9-1-76

Designation	Length (in.)	α of Dielectric (ppm/°C)	H_c (Oe)	B_r (gauss)
APS 169	5.145	15.4	3.73	655
APS 170	5.145	15.4	3.36	508
APS 171	5.145	15.4	2.90	649
APS 172	5.145	14.7	3.42	613
APS 173	5.145	14.7	3.41	666
APS 174	5.145	15.2	3.46	565

The data on H_c indicate fairly constant values, but the data on B_r show widely varying values, which do not correlate with dielectric or spray conditions. For example, sample APS 170 was sprayed onto the same dielectric as 169 and 171, yet B_r is about 25 percent lower for 170. Sample 174 also has an unexplainably low B_r. We will discuss these samples (170 and 174) further in Sec. 2.3.

APS samples 176 through 184 were sprayed on September 2. Eight of these nine samples were machined into full-sized phase shifters and annealed at 1010°C and 800°C before hysteresis measurements. Results of these measurements are summarized in Table VIII.

TABLE VIII

HYSTERESIS LOOP PROPERTIES OF APS SAMPLES PRODUCED ON 9-2-76

Designation	Length (in.)	α of Dielectric (ppm/°C)	H_c (Oe)	B_r (gauss)
APS 176	5.114	14.7	3.61	789
APS 177	5.145	14.7	3.41	633
APS 179	5.145	14.7	3.45	620
APS 180	5.145	14.7	3.28	590
APS 181	5.145	14.7	2.82	658
APS 182	5.145	14.7	3.18	631
APS 183	5.145	14.7	3.44	607
APS 184	5.145	14.7	2.40	274

The results of this series are certainly worse, in that very wide fluctuations in B_r (from 789 to 274 gauss) are observed. The similarity in spray conditions (Table III) and substrate expansion coefficient give no clue as to why this variation in B_r should occur. Sample 176 is the highest B_r which we have observed.

The APS runs on September 14, 15, and 21 used the dielectric type LMTF 195, where α = 15.25 ppm/°C. This material was close to the type LMTF 190, and had an expansion coefficient midway between the LMTF 190 and the LMTF 200 dielectric which was used extensively in earlier APS runs this quarter (See Table III). In these runs we were trying for a slightly denser ferrite coating, to achieve a larger B_r and phase shift. We attempted this by raising arc current and reducing the deposit rate, while maintaining the oven temperature as before. This approach did not give us results as good as the earlier runs, as shown by the low B_r in Table IX.

TABLE IX

HYSTERESIS LOOP PROPERTIES OF APS SAMPLES PRODUCED ON 9-14, 9-15, 9-21, AND 9-23-76

Designation	Length (in.)	α of Dielectric (ppm/ °C)	H_c (Oe)	B_r (gauss)
APS 186	5.145	14.7	3.21	480
APS 187	5.012	15.25	3.67	387
APS 189	5.145	15.25	3.08	517
APS 190	5.145	15.25	2.99	475
APS 192	4.905	15.25	3.36	568
APS 193	5.145	15.25	2.85	520
APS 195	1.815	15.25	3.80	668
APS 197	3.160	15.25	4.36	366
APS 198*	5.145	15.25	3.24	203
APS 199*	5.145	15.25	3.54	207
APS 200	1.696	15.25	2.43	697

* Samples had no anneal before measurements

Runs 185 through 194 used the LMTF 50 (G3) ferrite powder, whereas samples APS 195 and thereafter used the G4 type powder. The powder was dried and screened to minimize flow problems we had encountered with the G3 material. The continued increase in arc current throughout this series (in an effort to increase ferrite density) seems to have been counter-productive in terms of B_r. Samples APS 198 and 199 had no anneal after spraying and would be expected to have low B_r. The value of H_c is surprisingly low, indicating that considerable recrystallization has taken place at the high spraying temperatures. Typical values of H_c before annealing were in the 7-9 Oe range, reflecting the stresses present and the very fine-grained microstructure before annealing.

APS samples Nos. 201 through 210 were fired on the same dielectric composition at higher arc-current settings, lower powder-flow rates, and under faster deposit conditions. We now know (from the properties of earlier APS samples) that the spray conditions were probably too high in temperature.

Eight samples were sprayed in the final run (September 23). To reproduce the high $4\pi M_r$ of run APS 12, we increased the arc gas velocity and arc current, producing a net lower temperature. A new thermocouple arrangement in this run solved the controller problem. These latest materials have not been tested.

2.3 Dissection of Low Remanence Phase Shifters

Many of the phase-shifter samples discussed in Sec. 2.2 had low B_r, indicating these samples would not achieve the 340° phase shift required for the confirmatory testing. The B_r values were not only low but showed considerable variation from one sample to the next, with no apparent relation to dielectric composition or spray conditions. We decided to section to section two of the full-size phase shifters which had low B_r. The samples were APS 170 with B_r = 508 gauss and APS 174 with B_r = 565 gauss. These had been sprayed during a session when other samples having good hysteresis loop and microwave properties were produced.

Each of the samples showed reasonably uniform ferrite walls at the exposed ends (see End 1 and End 2 in Figs. 4 and 5. The 5.145 in. samples were cut into three equal segments which exposed two surfaces at the one-third distance (see 1/3, Fig. 4) and two surfaces at 2/3 the original length. For sample APS 170 the two dielectric halves showed 0.005 in. displacement at the 1/3 position and a severe nonuniformity in wall thickness. At the 2/3 position the wall was still nonuniform, the thin side remaining the same. The entire center segment evidently has one narrow and one thick wall, a condition which would be expected to produce a very low B_r. The final segment of APS, between the 2/3 location and End 2, has a nonuniform wall. The dielectric halves were still displaced 0.005 in. but were reasonably uniform at the ends. A similar dissection of sample APS 174 (Fig 5) showed similar wall nonuniformities, although not as extreme as APS 170

If we examine the machining process it will be evident why the ferrite walls appear uniform at the ends and can still be very nonuniform in the center. The machinist keys the grinding away of excess ferrite to the extreme ends of the sample where the bare dielectric rod extends beyond the ferrite coating. At the ends of the rod, then, assuming the machinist does his job, the ferrite coating around the dielectric is a uniform 0.050 in. These are the regions we see in cross section when the phaser is cut to its final length. Only destructive sectioning of the element would reveal the wall uniformities in the center regions.

Distortion or bowing of the samples could occur at any point in the high-temperature processing. We must follow through each phase, checking for straightness and correcting or changing the process to eliminate the sources. The holding oven where we store dielectric rods before spraying and composite samples after spraying has been held at 600° - 750°C. This high-temperature storage allows us to avoid thermal shock losses and keep sample transfer times down and sample production rate high. We may be forced to trade off these assets and lower the oven temperature if sample distortion occurs here. Certainly it would be preferable to avoid distortion by suspending the ceramic parts before and after spraying so that their

PBN-76-665

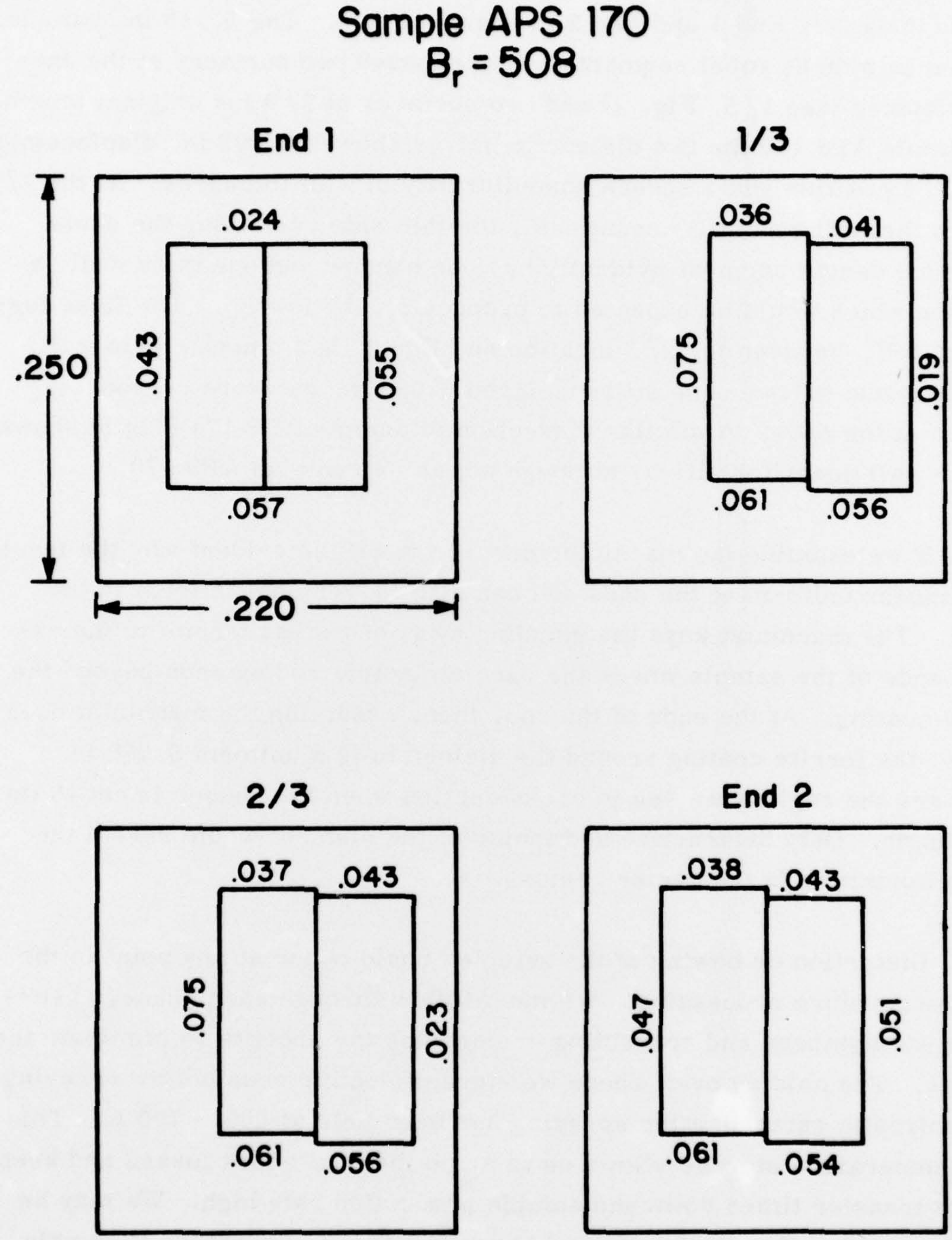


Figure 4 Cross Sections of Plasma Sprayed and Machined Phase Shifters. Wall thickness in inches as indicated

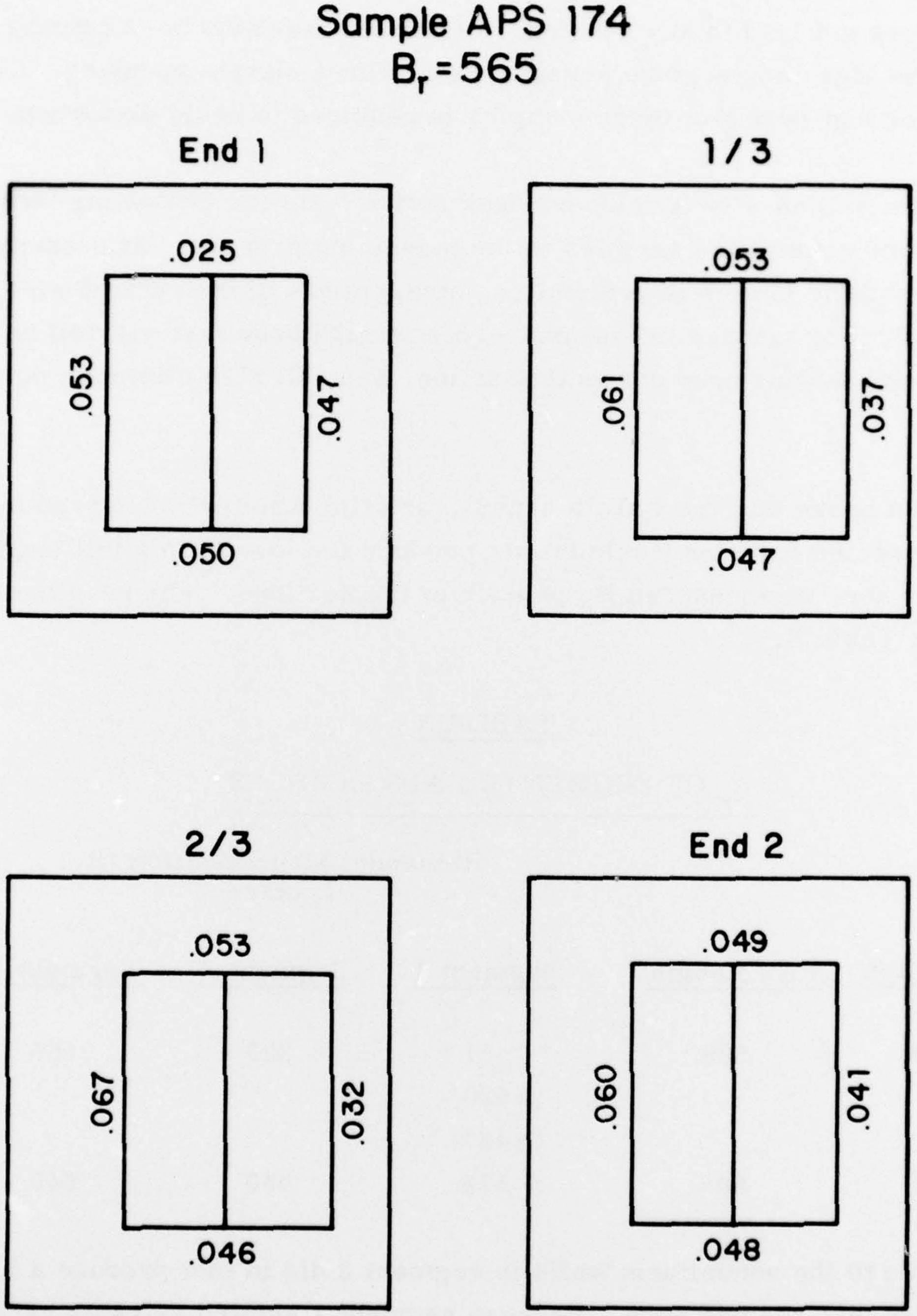


Figure 5 Cross Sections of Plasma Sprayed and Machined Phase Shifters. Wall thickness in inches as indicated

weight does not lead to any bending. Distortion may also be occurring during the high-temperature anneal which follows plasma spraying. Careful hanging or supporting of these samples is required to avoid distortion.

We will have to develop nondestructive tests for evaluating straightness before committing samples to the machining process. At present, the most promising avenue is determining straightness of the central wire slot using optical or mechanical means. This straightness test will tell us which steps in processing need corrective action, and will also eliminate poor samples.

To prove that the wall thickness variation which we observed in these dissected samples would in fact produce the low B_r in a full length phase shifter, we measured B_r on each of the sections. The results are shown in Table X.

TABLE X

B_r OF SEGMENTED APS SAMPLES

		Remanent Magnetization (B_r) (gauss)		
Designation	Full Length	Segment 1	Segment 2	Segment 3
APS 170	508	551	338	605
		a 629		
		b 467		
APS 174	565	575	550	640

For APS 170 the nonuniform walls in segment 2 did in fact produce a low B_r, lower than the two ends. Because segment 1 showed extreme variation on either end of the 1.7 in. piece, we decided to again section these pieces into equal segments a and b. We found the more uniform wall portion (a) near the original end had the expected higher B_r. Sample APS 174, with more uniform walls, again showed low B_r in the center segment where wall nonuniformity was the most extreme. There seems little room for doubt that wall variations are a major factor in the observed low B_r and its fluctuations.

3.0 CONCLUSIONS

During this quarter we have established the conditions for plasma spraying full-length samples at rates consistent with our production goal of five samples per hour. Over 100 samples have been evaluated, of which 39 were made into full length phasers and given appropriate heat treatment. One property that has been consistently low in the phase shifter samples is the remanent magnetization (B_r) and hence the microwave phase shift. Typical values have been 10 percent to 20 percent below the specified minimum of 340° phase shift ($B_r \geq 725$ gauss).

At the end of the quarter several phase shifters were sectioned and a major cause of the reduced B_r has been identified, i.e., variations in wall thickness of the ferrite resulting from shape distortion before the machining to external dimensions. This distortion is believed to take place at some time during the high temperature processing, i.e., before, during, or after APS deposition or during the high-temperature anneal which precedes machining. We will have to develop nondestructive tests of sample straightness to be applied at each step in processing to determine when this distortion takes place. The problem must be solved before the pilot production run begins.

4.0 PROGRAM FOR THE NEXT INTERVAL

The program for the next quarter will be to first identify the source or sources of sample distortion and then to devise techniques to avoid this problem. A part of this program will be to devise ways of measuring straightness after each processing step.

5.0 PUBLICATIONS

An abstract entitled "Plasma Spray Deposition of Composite Phase Shifters" was submitted and accepted for the Second International Conference on Ferrites, Paris, France. However, the problems on delivery of confirmatory samples made it impossible to spare the time and the paper was withdrawn.

6.0 IDENTIFICATION OF PERSONNEL

The personnel who contributed to this production development effort during the fifth quarterly reporting period, and the manhours worked by each is shown below. Biographies of these personnel have been supplied in previous quarterly reports.

Name	Hours
J. Green	7
J. Van Hook	146
L. Lesensky	2
D. Massé	10
H. Miller	12
R. Maher	416
Others	205
Total	798

DISTRIBUTION LIST

The Institute for Defense Analysis
Science and Technology Division
ATTN: Dr. Alvin D. Schnitzler
400 Army-Navy Drive
Arlington, VA 22202

Commander (2)
US Army Electronics Command
ATTN: DRSEL-PP-I-PI-1
(Mr. W. Peltz)
Fort Monmouth, NJ 07703

Dr. Joseph E. Rowe
Vice Provost and Dean
School of Engineering
Case Western Reserve University
312 Glennan Bldg.
Cleveland, OH 44106

Director, Applied Physics Laboratory
Sperry Research Center
ATTN: Dr. Richard Damon
Sudbury, MA 01776

Dr. Daniel G. Dow, Chairman
Dept. of Electrical Engineering
University of Washington
Seattle, Washington 98195

Commanding General
US Army Electronics Command
ATTN: DRSEL-TL-BM
(Mr. N.M. Wilson)
Fort Monmouth, NJ 07703

Commander
Harry Diamond Labs.
ATTN: AMXDO-RAA
(Mr. H.W.A. Gerlach)
2800 Powder Mill Road
Adelphi, MD 20783

Naval Electronics Laboratory Ctr
ATTN: Mr. E.D. Maynard, Jr.
Code 220
271 Catalina Blvd.
San Diego, CA 92152

Commander, RADC
Surveillance Technology Branch
ATTN: Mr. H. Chiosa, OCTE
Griffiss AFB, NY 13441

Director, National Security Agency
ATTN: Mr. A.T. Andrews, Jr. R335
Fort George G. Meade, MD 20755

Commander
US Army Production Equipment Agency
ATTN: AMXPE-MT (Mr. C.E. McBurney)
Rock Island, IL 61201

Advisory Group on Electron Devices (2)
ATTN: Working Group on Special
Devices
201 Varick Street
New York, NY 10014

Raytheon Company
Microwave and Power Tube Division
ATTN: L.L. Clampitt
190 Willow Street
Waltham, MA 02154

Dr. Turner E. Hasty, Director
Semiconductor, Research and Engr. Labs.
Texas Instruments, Inc.
PO Box 5013, M.S. 72
Dallas, TX 75222

Microwave Associates
ATTN: Dr. Joseph A. Saloom
Burlington, MA 01803

Commanding General
US Army Electronics Command
ATTN: DRSEL-TL-IM
(Mr. V.G. Gelnovatch)
Fort Monmouth, NJ 07703

US Army Ballistic Research Labs.
ATTN: Mr. D.G. Bauerle, AMXBR-CA
Aberdeen Proving Group, MD 21005

Director, US Army Ballistic Missile
Defense Advanced Technology Ctr.
ATTN: ATC-R, Dr. Bob L. Smith
P.O. Box 1500
Huntsville, AL 35807

Department of the Navy
US Naval Research Lab
ATTN: Mr. L. Whicker, Code 5250
Washington, DC 20390

Commander, AFAL
ATTN: AFAL/TE, Mr. J. Edwards
Wright-Patterson AFB, OH 45433

Lincoln Lab., MIT
ATTN: Dr. G.L. Guernsey
PO Box 73
Lexington, MA 02173

DISTRIBUTION LIST (CONTINUED)

AMPEX Corporation
ATTN: Mr. Gil Argentina 3-22
401 Broadway
Redwood City, CA 94063

US Army Missile Command
Directorate for Res. Engr. and
Missile Systems Lab.
ATTN: AMSMI-RLM
(Mr. P. Ormsby)
Redstone Arsenal, AL 35809

Microwave Applications Group
ATTN: Dr. Charles Boyd, Jr.
10021 Canaga Ave.
Chatsworth, CA 91311

US Army Elect Tech and Devices Lab(5)
ATTN: DRSEL-TL-MA
(Mr. Richard Babbitt)
Fort Monmouth, NJ 07703

NASA Headquarters
ATTN: Mr. C.E. Catoe
Code RES
Washington, D.C. 20546

Trans Tech, Inc.
ATTN: Mr. R. West
12 Meem Ave.
Gaithersburg, MA 20760

U.S. Army Missile Command
Director for Res. Engr. and Missile
Systems Lab.
ATTN: AMSMI-REI
(Mr. B. Spalding)
Redstone Arsenal, AL 35809

US Army Combat Surv and Target
Acq Lab
ATTN: DRSEL-CT-R
Fort Monmouth, NJ 07703

Defense Documentation Center (12)
ATTN: DDC-IRS
Cameron Station (Bldg. 5)
Alexandria, VA 22314