MASTER

DEVELOPMENT OF A LOW LOSS MAGNETIC COMPOSITE
UTILIZING AMORPHOUS METAL FLAKE

Third Semi-Annual Progress Report

For the Period

19 September 1979 - 18 March 1980

Metallurgy Laboratory

Corporate Research and Development

General Electric Company

P.O. Box 8

Schenectady, New York 12301

Principal Investigator: Lyman Johnson

Prepared for the

United States Department of Energy

Division of Electrical Energy Systems

Under Contract ET-78-C-01-3205

Project Manager: J.P. Vora

April 1980

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Foreword

The contributors to this project have been M.P. Borom, M.J. Curran, R.P. Laforce, H.H. Liebermann, L.C. Perocchi, A.C. Rockwood and J.R. Wilcox on helical ribbon and slot casting; and P.G. Frischmann and S.J. Kelly on magnetic measurements. All are members of the General Electric Corporate Research and Development Center.

I. INTRODUCTION

The objective of this project is to determine the feasibility of casting amorphous metal ribbon in the shape of a helix with properties suitable for motor applications. The tasks include 1) determination of the casting parameters required to produce a helical ribbon, 2) evaluation of magnetic properties and methods for bonding, and 3) developing methods for patterning a wheel for producing motor slots in as-cast ribbon.

It is expected that the resulting technology with further refinement and scale-up will provide a material with substantially lower magnetic losses than currently obtainable from electrical steels and will provide a low cost means of obtaining increased motor efficiency and opportunities for innovative motor designs.

To meet the project objective the work has been divided into three tasks whose technical approach is briefly described below.

Task 1: Helical Ribbon Casting

The planar flow casting methods previously developed for casting straight ribbon on the outer periphery of a casting wheel are being adopted and extended for casting helical ribbon. In order to produce a ribbon in helical form the casting is done on the flat side of the casting wheel as shown schematically in Figure 1. Optimization of the casting process is focusing principally on those aspects which differ from straight ribbon casting. These process parameter variables include nozzle geometry and positioning, wheel speed, surface conditioning and ribbon removal.

Task II: Property Assessment

The magnetic and physical properties are being measured and evaluated as a function of the processing conditions and annealing temperature. Physical

measurements include dimensional characteristics, surface and edge irregularities, ductility and degree of uniformity. Magnetic properties are established by measurement of the d.c. hysteresis loop. After reasonably good quality ribbon can be made, approaches for bonding the ribbon into a core-like structure will be evaluated.

Task III: Conductor Slot Casting

The objective of this task is to establish the feasibility of directly casting in the edge of the ribbon shapes simulating conductor slots. If this can be accomplished, no further punching or machining work would be required on the finished stator. Thus a relatively low cost finished product would result. The approach being taken to achieve the direct-cast conductor slots is to incorporate a low thermal conductivity pattern in the surface of the casting wheel so that this pattern creates a slow quenched, brittle pattern in the ribbon which can be broken out to create the conductor slots. This process is illustrated schematically in Figure 2. Three different approaches are being pursued to produce a pattern in the casting substrate that will provide a lower quench rate. These include: 1) a shallow grooved pattern with no filler material in the grooves, 2) a deep, narrow grooved pattern filled either with a low thermal conductivity, bonded glass or 3) filled with a low thermal conductivity, bonded metal.

II. PROGRESS THIS REPORTING PERIOD

II.1 Background

This section of the report covers the work during the third six-month period of the project. During the first year of the project (October, 1978 - October, 1979) the effort was directed toward developing methods for producing an amorphous metal flake composite for utilization in a motor. In that period, processing methods were established to produce good quality amorphous metal flake and to consolidate them into high density, stable compacts. However, it was discovered that the deformation-thermal cycle of the consolidation resulted in an intrinsic degradation of the magnetic properties to the extent that the compacts showed no promise of application in motors. Consequently, the technical approach was redirected toward the objective of establishing the feasibility of casting slotted helical ribbon for motor application.

II.2 Helical Ribbon Casting

Figure 1 shows schematically the basic approach to helical ribbon casting. By casting on the side of a rotating casting substrate, the cast product takes the shape of a helix. This shape is potentially ideal for use in motor stators. The simple helical shape without cast conductor slots could be used directly as the yoke of a motor stator. If conductor slots can be cast, then the entire stator would result.

During the first feasibility stage of helical ribbon casting, the background technology for casting straight ribbon has been applied. The important casting variables and the initial range of their investigation are:

Composition:	$\text{Fe}_{81.5}\text{B}_{14.5}\text{Si}_4$
Nozzle dimensions:	0.008-0.014 X 0.5-0.75 inches
Nozzle/wheel gap:	0.012-0.016 inches
Wheel material:	Copper and Copper-1% Chromium
Wheel finish:	600 grit paper plus wire brushing
Ejection pressure:	3-6 psi
Ejection temperature:	1250°C
Surface speed:	60-75 ft/sec
Casting atmosphere:	90 μ vacuum, 300 mm He, Air

Most of the casting runs were done at a diameter of 7 inches with 100 grams of material. This resulted in about 50 revolutions of casting.

The casting done in vacuum and helium produced ribbons of very good surface quality. However, the ribbon tended to leave the surface of the casting wheel within only a few inches of the nozzle. This was due to the combination of a large centrifugal force and low gas pressure holding the ribbon on the wheel. Since the ribbon departed so soon after becoming solid it was still quite hot. Figures 3 and 4 show the type of defect on the inside diameter of the ribbon which resulted.

In order to keep the ribbon on the wheel longer so that it quenches to a lower temperature, subsequent casting has been done in an air atmosphere. The casting equipment for these experiments is shown in Figure 5. In this condition we find the opposite problem: that is, the ribbon tends to stick to the wheel too long. If this happens it can stick a full revolution come back under the casting nozzle and ruin the casting trial.

To solve this problem in casting straight ribbon, we have found that by constantly conditioning the wheel surface with a rotating fine wire brush the departure point of the ribbon can be controlled. Additionally, the surface

conditioning from this rotating brush creates a steady state surface which gives a constant high quality ribbon surface.

With these basic casting methods and equipment in place about 20 casting runs have been run in air to debug the equipment and scope the important casting variables. Most of these runs have been made with a nozzle/wheel gap of 0.014 inches and a nozzle opening of 0.012 inches. Under these conditions we find that the edge of the ribbon tends to be somewhat rough. By flooding the area around the nozzle orifice with helium this condition can be improved but not completely eliminated. In future casting runs variations in the nozzle/wheel gap will be explored in hopes of eliminating this problem without the use of helium.

Another problem has been the frequent occurrence of producing wrinkled ribbon. This is particularly a problem with thicker ribbon. From past experience we know that this is due to deforming the ribbon at high temperature. In this specific case we think it is due to an early and irregular departure of the ribbon from the wheel. With early departure the ribbon is still quite hot - of the order of several hundred degrees centigrade. At these high temperatures the flow stress of the ribbon is quite low and it is easily deformed. If the departure point is irregular, then the bending associated with this irregularity causes the product to be wrinkled. Substantial work will be required to develop methods to control more precisely the ribbon departure point.

II.3 Property Assessment

The principal focus of property assessment is on the magnetic properties and their response to heat treatment. Because of the shape of the helical samples, special magnetic measurement and annealing fixtures have been made. Initially, the d.c. hysteresis properties of samples of about 1.1 revolutions are

measured as-cast and after a magnetic field anneal at 345°C . After further optimization of the processing has occurred larger samples will be evaluated for both d.c. and a.c. characteristics.

From the hysteresis loops the values of coercive force and flux densities at one and ten oersteds are tabulated for evaluation. Optimally, the coercive force should be as low as possible because it controls the losses. Coercive forces less than 0.05 oersteds are required to achieve the target losses. The flux densities at one and ten oersteds should be as high as possible, since they give an indication of the exciting field required. For motor applications the flux at ten oersteds should be greater than about 90% of saturation.

Table I shows the as-cast and annealed properties of some of the initial helical samples. As can be seen, there is a large variation in properties from sample to sample. This is to be expected at this stage in the development when many of the processing parameters are being varied. Significantly, some of the samples, e.g., 031080H2, have almost achieved the target goals.

TABLE I
D-C Magnetic Properties

Sample		$H_{\max}=1$ Oersted		$H_{\max}=10$ Oersted	
		Hc	B/B _s	Hc	B/B _s
022580H-1	As-Cast	.163	.31	.19	.72
	Annealed	.120	.49	.13	.95
022780H-1	As-Cast	.083	.36	.09	.73
	Annealed	.060	.56	.063	.95
030780H-1	As-Cast	.185	.15	.25	.52
	Annealed	.075	.67	.085	.90
030780H-2	As-Cast	.125	.18	.18	.55
	Annealed	.103	.35	.15	.82
030780H-3	As-Cast	.125	.20	.155	.63
	Annealed	.068	.69	.070	.93
031080H-1	As-Cast	.155	.17	.20	.59
	Annealed	.08	.65	.09	.87
031080H-2	As-Cast	.21	.19	.26	.54
	Annealed	.053	.78	.055	.88
031280H-1	As-Cast	.115	.21	.14	.60
	Annealed	.068	.69	.075	.90
Typical Straight Ribbon Sample					
030480WW2	As-Cast	.070	.46		
	Annealed	.042	.95		

II.4 Conductor Slot Casting

The approach being taken to directly cast conductor slots in the amorphous ribbon is shown schematically in Figure 2. The principal is to provide a parting line between the major portion of the ribbon which will serve as the finished stator stack and the ribbon cast in the conductor slot region which will be removed and recycled into the melt. The parting line is to be made by making a pattern in the surface of the casting wheel which has a lower thermal conductivity than the bulk of the casting wheel. Casting over this pattern region will result in a locally slower quench rate. The region in the ribbon that has the low quench rate will be significantly more brittle than the surrounding ribbon and should provide an easy parting line.

Three different methods for making the low quench-rate pattern are being pursued in parallel. These are 1) an unfilled shallow grooved pattern, 2) a grooved pattern filled with glass, and 3) a grooved pattern filled with a low thermal conductivity metal.

II.4.1 Planar Flow Casting Over a Grooved Pattern

An old and well-known method for the manufacture of narrow amorphous alloy ribbons is by the formation of a molten alloy jet from a pressurized nozzle and the subsequent impingement of this jet against a rapidly-moving substrate surface on which a continuous product is formed and chilled. A typical result of excessive substrate surface roughness or velocity in such a method is the spraying of molten alloy balls from the melt jet impingement area. The severity of fluid perturbation by a given substrate surface defect depends on the defect size, the substrate velocity, and the degree to which the alloy "wets" the substrate surface during casting. Because of the minimum substrate surface velocity which must be maintained during casting in order to result in an adequate molten alloy quench rate, only a certain maximum asperity size is

tolerable. For this reason, experiments in which segmented amorphous alloy ribbon or flakes were manufactured by this method in the first year of this contract were often plagued by a spray of molten alloy balls accompanying formation of the flake product. Such severe molten alloy perturbation during casting can be attributed to microtopological features of the insulator lines on the substrate surface used to define flakes. In these experiments, the nature of the surface asperity was typically a mismatch between interrupter line and substrate (either a fine crack-like separation or elevation difference at the surface) and/or a sticking of solidified material at the insulator line.

The planar flow method for casting wide metallic tapes directly from the melt relies upon a very small (~ 10 mils) crucible-substrate surface spacing in order to assure laminar melt flow during casting. The advantage of this casting method over conventional chill block melt-spinning lies in the reduced sensitivity to fluid flow perturbations by surface asperities on the substrate wheel. Because of the reduced sensitivity to substrate surface asperities, it may be expected that the detailed method of making the interrupter lines is not very crucial. As shown in Figure 6, initial experiments in the use of planar flow casting of patterned amorphous alloy tape were conducted by simply scribing the desired pattern with a sharp, conical tool bit. Both laminar melt flow and the presence of a melt perturbation attenuating "ceiling" are used to form a patterned tape without accompanying molten alloy ball spraying. A typical sample is in the form of a tape 1/2 inch wide and 0.0014 inches thick, having the substrate wheel's pattern visible on the surface. Since alloy which solidifies atop the grooves on the substrate surface is subject to a locally low cooling rate compared with the alloy in contact with the substrate material, a ductile product with local embrittlement along the pattern lines results. Separation of the cut-outs in the patterned tape is accomplished by simple manipulation of

the product after casting. Figure 7 shows a sample section of patterned tape from run 011680-1. The two platelets shown are typical for that particular run.

In further experiments, four types of patterns made up of grooves 6-8 mils deep were scribed into the surface of a 10 inch diameter water-cooled Cu-1 Cr substrate wheel. The patterns chosen were a series of rectangles with base at the tape edge, a series of triangles with base at the tape edge, a series of "teeth", and a simple geometric pattern totally within the ribbon surface, not touching the edges. In all cases, replication of the grooved pattern in the wheel was complete and residual deposits in or wear of the substrate grooves was minimal, even after 3000 cumulative feet of casting. Some regions of this ribbon can be seen in Figure 8.

Careful inspection of sample 011680-1 in Figure 7 reveals that the fracture line of the cut-out in the tape does not always follow the pattern line. This is because the tape itself was cast too thick to avoid overall mild embrittlement. Figures 9 and 10 are views of the upper surface of sample 020680-1 in the vicinity of the pattern lines. Both micrographs are included corners of the same rectangular pattern but were taken at different magnifications, as shown. Pattern lines parallel, perpendicular, and oblique to the casting direction in the tape plane shall be referred to as longitudinal,^(L) transverse^(T) and oblique^(O) lines. Note the good local definition of the L and T pattern lines and their intersection in Figure 9 whereas the longitudinal pattern line in the tape shown in Figure 10 extends beyond the scribe marks in the substrate surface. This undesirable "print-through" phenomenon appears to be the result of a delay in ceasing the extra melt flow in the L groove at the physical end of the longitudinal groove. Such print-through has mainly been found for large nozzle to wheel gaps, thicker tapes and deeper grooves, in which cases the extra melt flow would be more difficult to terminate at the end

of the longitudinal groove in the substrate surface. An experiment in which rectangular-patterned grooves of various depths were used revealed that only with grooves greater than 5 mils deep did print-through occur for amorphous ribbon made about 1.8 mils thick.

Metallographic inspection has revealed the L and T pattern lines to be quite different in cross-section, as shown in Figures 11 and 12. Support is given to the view that there is established a smooth melt flow at longitudinal grooves by the smoothness, uniformity and symmetry of the L line cross-section in Figure 11. Close inspection of this micrograph reveals tiny crystallites which have nucleated and grown at the center of the L line, these being largely responsible for the local embrittlement found. Note the decrease in local sample thickness as one approaches the center of the L line cross-section. This occurs because of the local "drooping" of the L line material into the groove and results from constant melt flow rate across the width of the tape being cast.

The embrittled T line cross-sections are quite different from those of the L lines, as seen in comparing Figures 11 and 12. The geometric uniformity observed in the L lines is lacking in the T lines, where there is evidence of molten alloy turbulence at the T groove. This apparent melt turbulence caused by the impact of the T groove on the liquid reservoir between the crucible and substrate surface has been successfully attenuated by virtue of planar flow casting. No molten alloy balls expected to be cast as a consequence of such turbulence were observed during the run. However, the impact of the T groove may cause poorer contact between tape and substrate immediately following the T groove, sometimes resulting in oxide discoloration and severe tape embrittlement immediately following the T groove. The greater turbulent character of the T groove is further reflected by the diffuse, irregular

crystallization pattern observed in the T line cross-section in Figure 12. It is presumed that the generally broader crystallized and embrittled zone in the T grooves makes cut-out separation somewhat easier along these than along L grooves where the crystallization/embrittlement zone is much narrower.

While the initial success of planar flow casting over unfilled grooves has been good, several problem areas have been identified. First, the degree of embrittlement in the longitudinal lines is not very great and it is thus somewhat difficult to achieve clean separation of the slot segments. On the other hand, because of the high turbulence following the transverse groove the brittle crystallized region is quite wide. This allows easy separation but may lead to poor magnetic properties in the remaining teeth.

Secondly, as can be seen in Figures 11 and 12 the ribbon is somewhat thicker near the pattern lines than far away regions. This may cause serious problems in stacking the stator core if it is not improved.

However, in general these preliminary results are quite encouraging, and this approach will be pursued further.

II.42 Glass-Filled Slots for Casting of Patterned Amorphous Metal Ribbon

In order to avoid some of the problem associated with casting over unfilled grooves discussed in the previous section another basic approach is to provide a pattern of fine grooves filled with a material of low thermal conductivity in the copper casting wheel. Several low thermal conductivity materials have been tried with varying degrees of success in providing a discontinuity in the quenching rate of the amorphous metal ribbon. The materials include epoxy resin, stainless steel strips and glass. The current section deals only with the results of the application of glass to grooved copper substrates.

In the last semi-annual report it was demonstrated that bubbles intersecting the surface of glass placed in slots in the copper quenching wheel totally disrupted the flow of the molten metal and, therefore, must be eliminated or at least considerably reduced in both size and number. It was recognized that the bubbles were derived from at least two different sources during the enameling process which involves the application of finely powdered glass to the metal substrate. The first source is from gas which becomes entrapped in the coating as the glass surface fuses over. The source of the entrapped gas is either adsorbed gases on the surface of the particles or gaseous decomposition products from the enamel mill additions. A second possible source of bubbles in the glass is from gas, most likely hydrogen, evolved from the OFHC copper.

The entrapped gas problem was attacked in the last semi-annual reporting period by fusing bubble-free glass fibers in the slots and by immersing slotted copper plates in crucibles of molten glass. Large bubbles, apparently still emanating from the substrate, prevented success. An unsuccessful attempt was made to eliminate the substrate as a source of gas by vacuum annealing the copper samples at temperatures up to 700°C for as long as 30 min. In the current reporting period vacuum anneals at 900°C for 5 hours were similarly unsuccessful.

Work during the current reporting period was directed at optimizing the enameling technique by varying the firing parameters of time and temperature and evaluating alternate glass compositions.

Preparation of Enamel Slips

Thirteen glasses listed in Table II (12 from commercial sources) were each ground to -120 mesh and individually suspended by agitation in methanol at a solids loading of 30 v/o. The use of conventional mill additions such as gum

tragacanth and clay was avoided in order to minimize enamel outgassing during firing. The slip was applied either by spraying or by spreading with a spatula with the intent of providing a surface coating no thicker than .010". In one experiment, a button of bubble-free glass was applied directly to the copper substrate. The enamel coatings were fired on the copper substrates at temperatures between 500 and 900°C for times ranging from 3 to 30 min.

TABLE II
Designation of Enamel Frits Evaluated
(listed by supplier)

<u>GE/CRD</u>	<u>O'Hommel</u>	<u>H. Thompson</u>	<u>Glidden/Pemco</u>
			3316+
2042+	901+	706+	H-3638+
	1625+	969+	H-7213+
			I-413*
			I-1452*
			J-318+
			Mirac-5001**
			U-9867+

*blue, ground-coat frit

+clear cover coat enamel

**clear, crystallizing

Copper Substrate Preparation

The copper used was OFHC 1/32", 1/16", 1/8", 1/4", 3/8" and 1/2" thick. The bulk of experiments were made on 1/16" thick 2" x 2" plates. Prior to coating, different copper samples were prepared in a variety of ways; i.e., washed to remove grease and scale, bright dipped, etched with nitric acid, vacuum annealed at 650, 700, and 900°C, nickel plated, and pre-oxidized.

Most of the enameled samples were unslotted but several contained slots (10 mils deep by 5, 10, and 20 mils wide and 10 mils wide by 5, 10, 20 and 50 mils deep. Most slots were saw cut but some samples had circular slots made by electrodischarge machining.

Results and Discussion

It was found that pretreating the copper substrate did very little to improve the quality of the coating. Adherence was reduced with the application of a nickel plate with no decrease in the bubble content. Vacuum annealing of the copper reduced the number of bubbles formed, as is indicated in Figure 13 for enamel J318, but the size and number of bubbles was still too great for producing acceptable parts. The pretreatment of the copper was, therefore, reduced merely to washing away the surface grit and grease.

The appropriate firing schedule for each enamel depends on the specific temperature/viscosity relationship of the glass and must be experimentally determined for each enamel. In general, if the enamel is too refractory, too much oxidation of the copper will occur before the enamel fuses over and isolates the copper/glass interface from further atmospheric oxidation. The commercial enamels have been developed to mature in an appropriate temperature range. After fusing, the enamel must reach a temperature at which its viscosity is sufficiently low to allow bubbles from entrapped gases to evolve

from the coating within the firing time. The translation of viscosity into firing parameters is a trial and error process.

The progression of bubble formation and development during various firing cycles is illustrated in the photomicrographs taken normal to the enamel surface using oblique lighting in Figures 13, 14 and 15 for three different enamels. Figure 13 shows that for enamel J318 a profusion of bubbles remains in the coating after firing for only 5 min. at 600°C. The bubbles are assumed to originate principally from retention of gases evolved from the surface of the glass particles or trapped in the interstices between the particles as the surface fuses over. Increasing the time at 600°C decreases the number of bubbles but they increase in size reaching in the 30 min. firing a diameter almost exceeding the proposed .010" slot width. The second stage bubble development is assumed to be due to coalescence of smaller bubbles and evolution of additional gas from the metal substrate.

The bubble structure developed in J318 at 700°C (see Figure 13) at 3 min. and 6 min. is almost identical to that developed at 600°C at 15 min. and 30 min., respectively, illustrating the role of temperature in the kinetics of bubble removal and development. Also shown in Figure 13 is that, even after firing the enamel for 30 min. at 700°C, a copper substrate which has been vacuum annealed for 1 h at 650°C still continues to generate unacceptably large and numerous bubbles.

Figure 14 shows an example of the development of bubbles in enamel Mirac 5001 which undergoes crystallization during firing. At 700°C, 2 mil dia. bubbles are the maximum size observable in a gossamer network of needle-like crystallites. Increasing the firing time to 15 min. increases the development of the crystals to the point that they begin to obscure the bubbles but some bubbles with a diameter of 3.5 mils can be seen. A true assessment of the

bubble structure of an opaque coating can only be gained by inspecting a metallographic cross-section. Increasing the firing parameters to 800°C for 6 min. results in larger, more numerous but less objectionable bubbles than those observed for enamel J318 in Figure 13.

The most encouraging results were obtained with frit I413 as illustrated in Figure 15. Only relatively isolated, 2 mil dia. bubbles are observed on firing at 700°C for 6 min. and the bubbles increase to only 3.5 mil in dia. on extending the firing to 15 min.

The key to controlling the bubble structure was demonstrated by the experiment shown in Figure 16, a photomicrograph taken normal to the copper surface, in which a button of bubble-free J318 glass was placed on a .060" thick copper substrate and fired for 2 min. at 900°C. The glass button first softened and conformed to the copper substrate then slumped and spread. The region of first and longest contact between the copper and the softened glass shows in Figure 16a as the circular, bright area which is shown in higher magnification in Figure 16b. The bright area is shown to contain many bubbles greater than 10 mils in diameter. The glass in the bright area is in direct contact with the metal without the presence of an intermediate oxide layer. The region beyond the initial contact area still contains an intermediate oxide layer and the glass, as is shown in higher magnification in Figure 16c, is essentially free of bubbles.

It can be concluded from Figure 16 that the intermediate copper oxide serves as a barrier to the evolution of gas from the OFHC copper. (Since OFHC copper has been processed in hydrogen and consequently contains large amounts of dissolved gas, an improvement in enamel quality may be obtained by utilizing conventionally processed copper at the expense of some loss in thermal conductivity.) A correct enameling schedule, therefore, must provide the formation of sufficient oxide to serve as a barrier to substrate outgassing

during the period in which entrapped gas is being eliminated from the enamel and the enamel is concomitantly dissolving the substrate oxide. Such a schedule is approached in the firing of frit I413 on a .060" thick copper sheet for 6 min. at 700°C. The retention of a discrete oxide layer between the glass and the copper may adversely affect the glass/metal adherence. Additional latitude may, however, exist over that for flat sheet applications in that the glass in slots is also mechanically held in compression by the metal.

The attainment of a perfect process is hampered by imperfections in the oxide coating. Examples of breaks in a continuous oxide coating giving rise to the evolution of gas from the substrate are shown in Figure 17. The correspondence between the bubble in the glass and the break in the coating is clear.

The most bubble-free coatings were previously achieved with enamel layers less than 10 mils thick. Bubble retention was observed to increase with increasing enamel thickness, therefore, there was some concern regarding successful filling of 50 mil deep slots. Slots deeper than 10 mils were desired for greater latitude in subsequent metal-surface-dressing operations. In spite of the shortcomings of oxide discontinuity and long degassing paths in the slots, successful coatings with a low incidence of bubbles were achieved in the full range of width and depth of slots attempted including slots 10 mils wide by 50 mils deep. Examples of slots in 60 mil thick copper sheets filled with I413 fired for 6 min. at 700°C are shown in the metallographic cross-sections of Figure 18. The 50 mil deep slots required two enamel firings for complete filling.

In anticipation of enameling casting discs .5 inches thick, enameling parameters were evaluated for substrate test sample thicknesses of .062, .125, .250, .375 and .500 inches. Successful, low-bubble-content enamel coatings were achieved by inserting the test samples into a 700°C furnace and holding

the .125, .250, .375 and .500 thick pieces for 6, 8, 15, and 15 min., respectively. A plot of temperature vs. time was obtained for each specimen by embedding the tip of a chromel-alumel thermocouple in a hole drilled in the copper. The temperature profiles will be used as a guide in adjusting firing conditions for larger specimens and different furnaces.

All of the candidate glasses were tested for erosion resistance by squirting a small, low velocity jet of molten alloy (~5 gms at 4 psi through a 20 mil orifice) onto the enameled surface of a copper plate. Pemco I413 appears to have a slightly poorer resistance to erosion by the impinging jet than do several of the other glasses. However, this test subjects the glass to a more severe environment than does the actual casting in which the melt impinges intermittently and the heat is rapidly removed by the adjacent copper.

Assuming the glass will withstand the less severe casting environment and that the lack of porosity is the most critical criterion, Pemco I413 will be used for the first actual casting experiments. A flat copper wheel for the casting of helical ribbon is being prepared with glass filled grooves. The optimum firing cycle for minimum porosity will be determined by several trials on copper with the actual wheel dimensions. The wheel has a variety of groove depths to determine both the maximum filling depth of the glass and the minimum glass thickness required for good thermal insulation during casting. Presently these grooves are being mechanically scribed into the copper but tests are being conducted to determine the effect of grooving by electro-discharge machining (EDM). EDM charges the copper with hydrogen and could possibly increase the amount of glass porosity. Actual casting experiments on this glass filled wheel will determine: the amount of local ribbon embrittlement above the glass; the ease of cut-out separation; the tendency for glass-metal sticking; and the resistance of the glass to erosion and thermal cracking.

II.43 Metal-Filled Slots for Casting Patterned Amorphous Metal Ribbon

The use of stainless steel as a groove filler material is also being investigated. Preliminary casting runs on stainless steel inserts did not result in complete separation of the ribbon over the insert but did have a region of embrittled material. The difference in erosion resistance between the stainless steel and the copper may become a problem during extended runs. Figure 19 shows a profilometry trace of the wheel surface after being cast on for about 500 revolutions. The copper appears to erode faster than the insert or the insert may be moving out of the slot forming a step on the surface of the wheel. These steps cause splashing of the molten alloy during casting. However, planar flow casting will be used in future runs instead of the molten alloy jet casting used here. Because of the reduced turbulence and impact velocity in planar flow casting the flow casting step problem should be somewhat alleviated. Future use of a surface conditioning wheel should also help to ensure a smooth casting surface.

Although all casting to date has been done on a wheel with copper plated stainless steel inserts, attempts are being made to fill the grooves with a low thermal conductivity metal by plasma spray. This is in anticipation of future groove patterns which may be too intricate for practical use of inserts. Complex patterns could be put into the wheel by EDM then easily filled with a plasma spray. The excess sprayed metal would then be ground down to the copper substrate exposing the filled grooves. Initially, several test plates with a variety of grooves will be plasma sprayed with a commercially available nickel chromium powder. These will determine the strength of bonding and the resistance to interfacial cracking during grinding of the surface. Assuming this will be satisfactory, a wheel is being constructed for pattern casting. It will

then be possible to cast sufficient ribbon to determine the effectiveness of the metal filled pattern in causing local embrittlement.

Figure 1. Schematic representation of helical ribbon casting

Figure 2. Schematic representation of conductor slot casting in a helical ribbon.

Figure 3. Vacuum cast ribbon with inside diameter defects.

Figure 4. Helium cast ribbon with inside diameter defects.

Figure 5. Air casting equipment

Figure 6. Planar flow casting over a pattern

Figure 7. Directly cast cut outs using planar flow casting over unfilled shallow grooves

Figure 8. Directly cast cut outs using planar flow casting over unfilled shallow grooves

Figure 9. Ribbon cast over unfilled shallow grooves. Ribbon casting direction is to the right (50X)

Figure 10. Ribbon cast over unfilled shallow grooves. Ribbon casting direction is to the right (25X)

Figure 11. Cross section of a longitudinal pattern line in a cast ribbon. (200X)

Figure 12. Cross section of a transverse pattern line in a cast ribbon. (200X)

600°C

a) 5 min.

b) 15 min.

c) 30 min.

700°C

d) 3 min.

e) 6 min.

f) 30 min. (after 1h vac. anneal)

Figure 13. Photomicrographs taken normal to the surface of enamel J318 on copper illustrating the bubble structure developed on firing at 600°C for a) 5, b) 15 and c) 30 min. and at 700°C for d) 3, e) 6 and f) 30 min. The OFHC copper for sample (f) had been annealed in vacuum for 1h at 650°C. (50X)

700°C

a) 6 min.

b) 15 min.

800°C

c) 6 min.

Figure 14. Photomicrographs taken normal to the surface of enamel Mirac 5001 on copper illustrating the bubble structure and crystal formation developed on firing at 700°C for a) 6 min. and b) 15 min. and at 800°C for c) 6 min. (50X)

700°C

a) 6 min.

b) 15 min.

Figure 15. Photomicrographs taken normal to the surface of enamel I413 illustrating low bubble content after firing at 700°C for a) 6 min. and b) 15 min. (50X)

a) Entire zone (5.2X)

b) Oxide-free center area (17X)

c) Area containing undissolved oxide (17X)

Figure 16. Photomicrographs taken normal to the surface of a bottom of initially bubble-free J318 glass fired on OFHC copper at 900°C for 2 min. showing in a) the entire glass/Cu contact area, in b) the high bubble concentration in the initial contact area and in c) the low bubble content where undissolved oxide remains. ((a) 5.2X, (b) and (c) 17X)

a) Glass U9867, 700°C-6 min.
(note irregularities in oxide thickness)

d) Glass I413, 700°C-6 min.
(note bubbles above breaks in oxide layer)

b) Glass U9867, 700°C-15 min.
(note bubble formation at break in oxide)

e) Glass H7212, 700°C-6 min.
(note small bubble forming on wall of slot at break in oxide)

c) Glass U9867, 800°C-6 min.
(note total dissolution of oxide layer)

Figure 17. Photomicrographs of metallographic cross sections of Cu/glass interface showing in a-c the progressive loss of the intermediate copper oxide layer and emergence of gas from the oxide-free regions of metal and in d and e gas bubble formation at breaks in the oxide film. (1000X)

a) Slot $\sim .008'' \times .020''$ deep

b) Slot $\sim .020 \times .006''$ deep

c) Slot $\sim .008'' \times .010''$ deep

d) Slot $\sim .010'' \times .050''$ deep

Figure 18. Photomicrographs of metallographic cross-sections showing slots in copper plates filled with I413 glass applied by firing at 700°C for 6 min. Fifty mil deep slot in (d) required two applications of enamel for complete filling. Glass in all slots contains only small and infrequent bubbles. (a-c (200X), d (81X)).

Figure 19. Profilometer trace of copper wheel surface containing stainless steel insert after 500 casting revolutions.