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FINAL REPORT

to

LAWRENCE LIVERMORE LABORATORY
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From
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Introduction

This report covers the work by Supercon, Inc. on the development of manufacturing techniques for a multifilamentary Nb_3Sn composite conductor of unique design during the period from October 7, 1976 to September 30, 1977.

The necessity for magnetic containment of the plasma for producing fusion power provided the incentive for this work. The goal is the development of procedures for the fabrication of a conductor capable of carrying 10,000 amperes in a 12 tesla field.

The more common techniques for producing a multifilamentary Nb_3Sn conductor involve the manufacture of a conductor containing niobium filaments in a bronze or copper matrix. In the case of the copper matrix, the conductor is coated with tin and heat treated to diffuse the tin through the copper to the niobium where the Nb_3Sn is formed on the surface of the filaments. Because of the need to diffuse the tin all the way through the conductor uniformly, this technique is limited to small wires of less than 0.3mm diameter. Heat treatment of the bronze matrix conductor likewise causes diffusion of the tin from the bronze to the niobium. Because the bronze surrounds each filament there is no limit on the size of this style of conductor. The bronze is usually a simple binary alloy of copper with 10 to 13.5 weight per cent tin. Others have studied the effect of small amounts of a third element. Both techniques have a serious drawback in that the residual tin in the matrix causes the matrix to be highly resistive and reduces the stability of the conductor. Copper must be added either externally or as little "islands" in the matrix and must be surrounded with a suitable barrier material to prevent contamination by the tin.

The uniqueness of the Supercon approach lies in the use of hollow niobium filaments in a high conductivity copper matrix. Each filament contains a core of bronze. During heat treatment the tin migrates from the bronze to the inner wall of the niobium filament where the Nb_3Sn is formed. The outer unreacted part of the niobium filament serves as a barrier to protect the purity and high conductivity of the copper. Thus, each filament is stabilized in the same way as in the composite NbTi conductors.

During this period, test samples of conductor containing large numbers of filaments were made from small two inch diameter billets. The samples were given three different heat treatments and tested by Lawrence Livermore Laboratory. This data is included in this report. An effort to make a test piece of larger size from eight inch billets was unsuccessful. Failure appeared to be due to faulty temperature control during heating for extrusion. An effort to repeat this experiment was hampered by difficulty in obtaining more bronze. The long delay prevented a further attempt during this period.

Section II

Experimental Work

1. Two Inch Diameter Billets

Two 2" diameter primary extrusion billets were made. Material from these was used to fabricate four different secondary extrusion billets. These four had different numbers and sizes of filaments. Descriptive information is presented in Table I.

No difficulties were encountered in the manufacture of conductor from these extrusions. Two sizes were made for short sample test, 0.160" diameter round and 0.065" x 0.200" rectangle.

Samples were given heat treatments at either 750°C for 36 hours, 700°C for 100 hours or 650 C for 150 hours. These were tested at Lawrence Livermore Laboratory and previously reported.(1) The test results are reproduced in Figures 1 to 3.

2. Eight Inch Diameter Billets

Following the successful completion of conductor from the 2" diameter billets, an 8" diameter primary billet was made followed by an 8" diameter secondary extrusion. Descriptive information concerning these billets is presented in Table II. Comparative information on one set of the 2" diameter billets is included. The primary 8" diameter extrusion was successful and wire has been taken down to 0.035" and 0.027" diameters and I-H tested successfully. A seven strand cable made from the 0.027" diameter wire was sent to Lawrence Livermore Laboratory for test. A 0.027" diameter wire has filaments of roughly the same size (36 μm) as does the final product of the double extrusion. The second 8" diameter billet apparently extruded successfully but when the extruded rod was examined, many irregular and broken filaments were seen. Comparison of the two sets of billets in Table II shows one that only two factors varied appreciably between the two inch and eight inch billets. The two inch billets had a substantially higher percentage of copper and a lower extrusion reduction ratio. These conditions were true for both the primary and secondary extrusions. Inasmuch as the primary eight inch extrusion was successful, it is questionable whether these factors contributed to the failure of the eight inch secondary extrusion.

(1) Letter from Jon Zbasnik,
Lawrence Livermore Laboratory
dated May 31, 1977

The failure of the secondary 8" diameter billet may have been due to inadequate heating. If one examines the force required to extrude the primary and secondary billets, the primary required 3370 tons at a temperature of 655°C; the secondary required 3750 tons at a temperature of 643°C. As the temperatures were about the same, the secondary extrusion should have run at a lower tonnage as it had 20% more copper than did the primary and copper extrudes much more easily than does bronze or niobium in this temperature range. A further indication that the controls on temperature were not acting properly was that a niobium-titanium billet extruded at the same time took about 8% more tonnage than other billets of the same design extruded at the same temperature at other times. Examination of the cross section of the extruded rod shows a higher percentage of the filaments are badly torn near the center of the rod. One may argue that these did not deform as uniformly as did the outer ones because they were at a lower temperature.

Another possibility is that the vacuum in the billet was lost because of damage to the billet in a fall during shipping. It was examined following the fall and no cracks were detected using a die penetrant method of inspection, but a slow leak may have developed nevertheless, especially during billet heating.

Despite the poor appearance of the extruded rod, samples were drawn to the test size, 0.2 inch x 0.4 inch and heat treated for 52 or 143 hours. Tests of these samples supported the early conclusion that this rod's filaments were badly fractured.

Wire samples from the preliminary 8 inch extrusion were drawn to 0.035 inch diameter (45 μm diameter filaments), and heat treated for various times ranging from 6 to 174 hours to determine the effect of heat treatment time on the current density produced in the wire. The results are shown in Figure 4. The wire was tested in a hairpin configuration. It was bent into the hairpin before the heat treatment. The tests show an increase in current density up to about 100 hours. Beyond that time, values at 10 T or lower tend to decline whereas at 12 T or above the curve is relatively flat.

Section III

Future Work

Initially, we proposed to successfully scale-up from two inch diameter to four inch diameter billets. This is only a four times scale-up from the successful two inch diameter billets and permits a much less expensive and faster technique for examining the variables. Eight inch diameter billets will be tried only if the four inch scale-up is successful. In addition, an experiment will be run which, if successful, should substantially reduce the cost and simplify the manufacture of the conductor. In the current process, niobium tubes are procured. A rod of bronze is placed in each tube and the material is co-reduced to a size for use in the multifilamentary extrusion billet. In the proposed technique, less expensive niobium sheet is procured. This sheet is wrapped around a cylinder of bronze, and both are placed in a copper extrusion can and extruded to give rod stock for use in assembling multifilamentary composite extrusion billets. Besides simplifying one manufacturing step, an advantage is possible by using a rolled sheet texture as opposed to the texture in the tubes to obtain superior and more uniform wall thickness.

The basic idea of using a tubular barrier element, such as niobium, in a high conductivity copper matrix offers other possible combinations. These could be the use of powder metallurgy methods to the use of "in-situ" type Nb_3Sn, V_3Ga cores which have received much attention recently. Since Lawrence Livermore Laboratory has supported the tubular approach for many years, it would be logical to continue this support.

TABLE I

Summary of Data on 2 Inch Double Extrusions

Primary Extrusion Billets Identified 256XE-32 and 33

Billet Diameter	2"
Die Diameter	0.425"
Extrusion Reduction	23X
Extrusion Temperature	620°C
No. of Filaments	37
Copper:Filament	1.1:1
Bronze:Niobium	1.4:1

Secondary Extrusion Billets

Billet Diameter	2"
Die Diameter	0.425"
Extrusion Reduction	23X
Extrusion Temperature	620°C

	No. of Filaments	Copper: Filament	Average Filament Size at .065" x .200"
256XE-34	703	4:1	55 μm
-35	1369	4.4:1	38 μm
-36	1147	4:1	43 μm
-37	2257	5:1	28 μm

TABLE II

Comparison of 2 Inch and 8 Inch Billet Experiments

	2" Billet	8" Billet
Identity	256XEE-37	281XEE-1
No. of Filaments	2,257	58,081
Pattern	61 x 37	241 x 241
Size of Filament	28 μm	23 μm
Size of Sample	0.065" x 0.200"	0.2" x 0.4"
Copper:Filament Ratio	5:1	0.82:1
J_c at 12T/Filament (HT at 750°C)	1.8×10^4 A/cm² (36 hrs at Temp.)	-
Primary Extrusion		
Identity	256XE-33	281XE-1
Temperature	1150°F	1200°F
Heating Time	2 Hours	3.5 Hours
Reduction	23X	64X
Die Diameter	0.425"	1.0"
Size of Filament in Billet	0.195"	0.409"
Size of Filament as Extruded	0.041"	0.051"
Secondary Extrusion		
Temperature	1150°F	1200°F
Heating Time	2.5 Hours	3 Hours
Reduction	23X	64X
Die Diameter	0.425"	1.0"
Drawing Reduction Between Extrusions	5.4X	5.2X
Size of Filament in Billet	440 μm	576 μm
Size of Filament as Extruded	92 μm	70 μm
Drawing Reduction to Sample	10.9X	9.8X
Bronze:Niobium in Filament	1.4:1	1.5:1

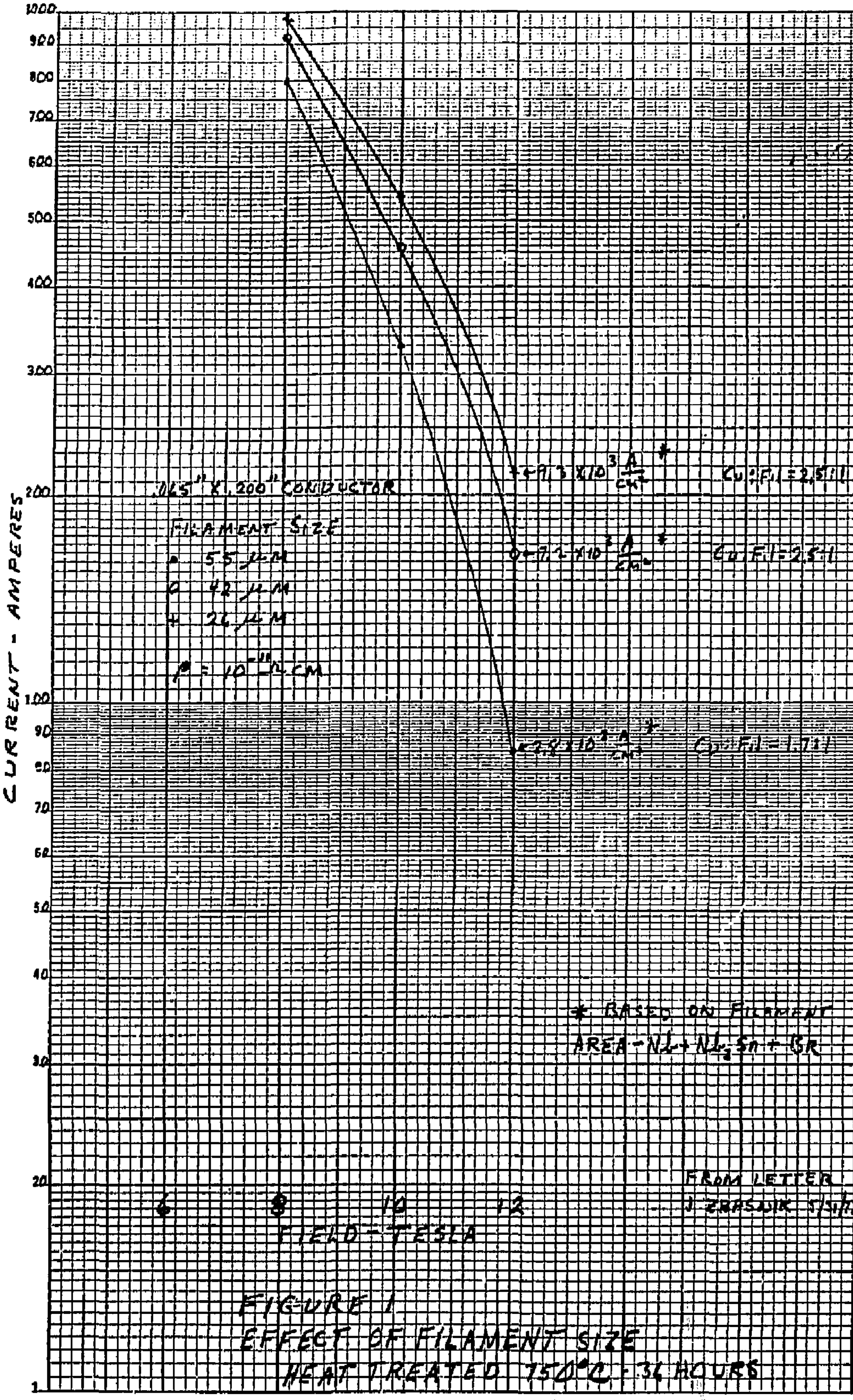

FIGURE 1
EFFECT OF FILAMENT SIZE
HEAT TREATED 750°C - 36 HOURS

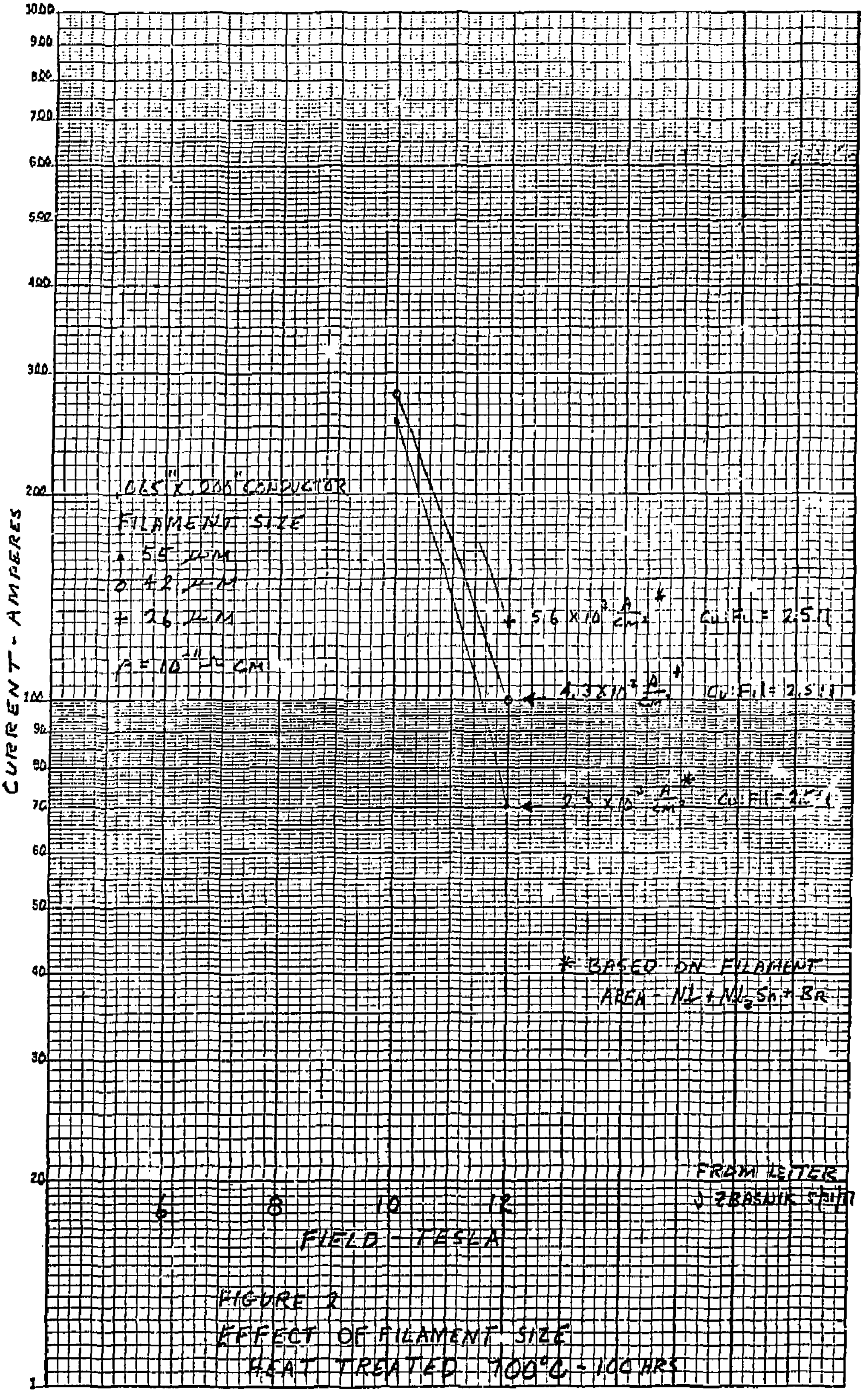

FIGURE 2
EFFECT OF FILAMENT SIZE
HEAT TREATED 700°C - 100 HRS

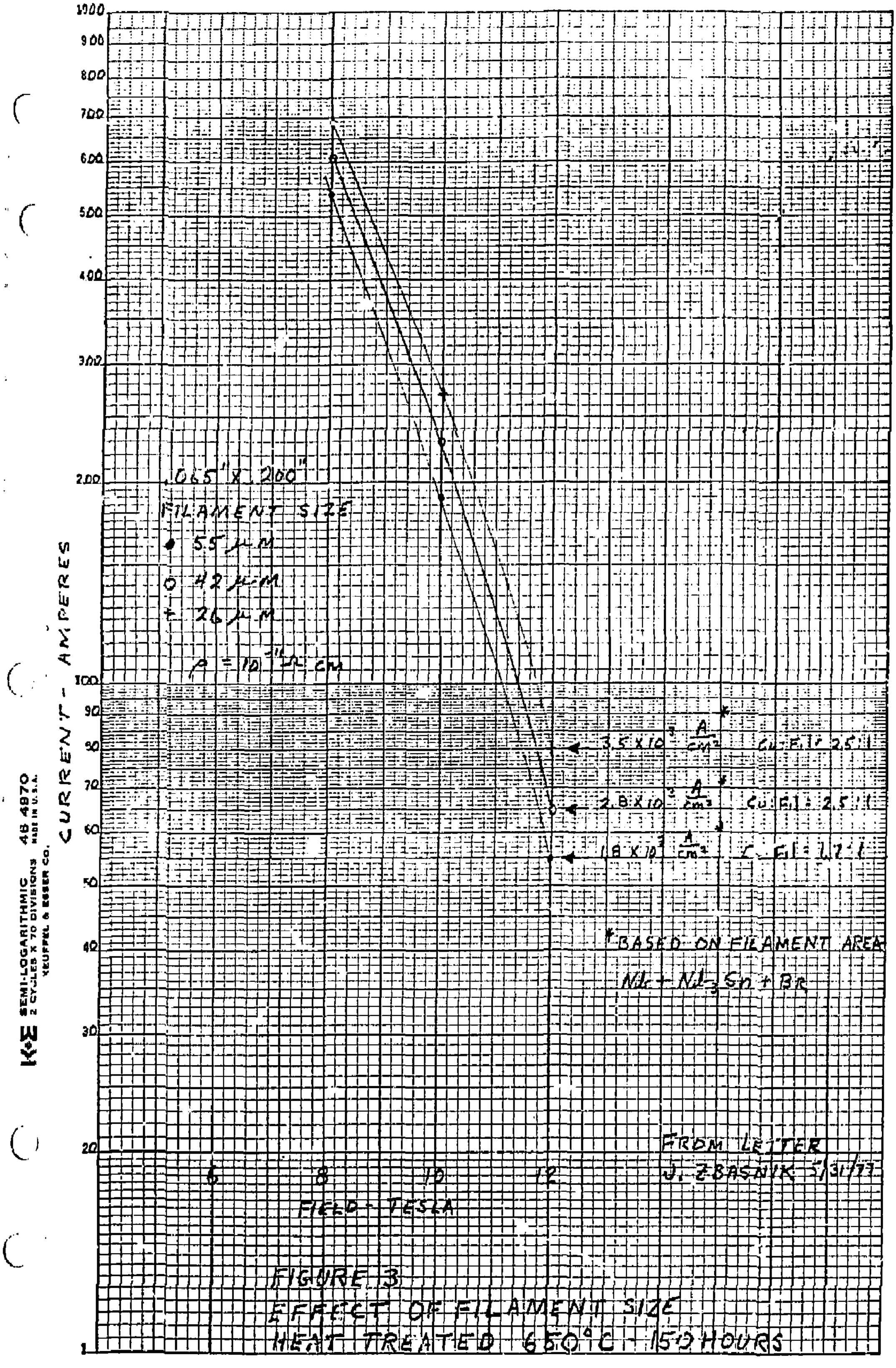

FIGURE 3
EFFECT OF FILAMENT SIZE
HEAT TREATED 650°C - 150 HOURS

236,997

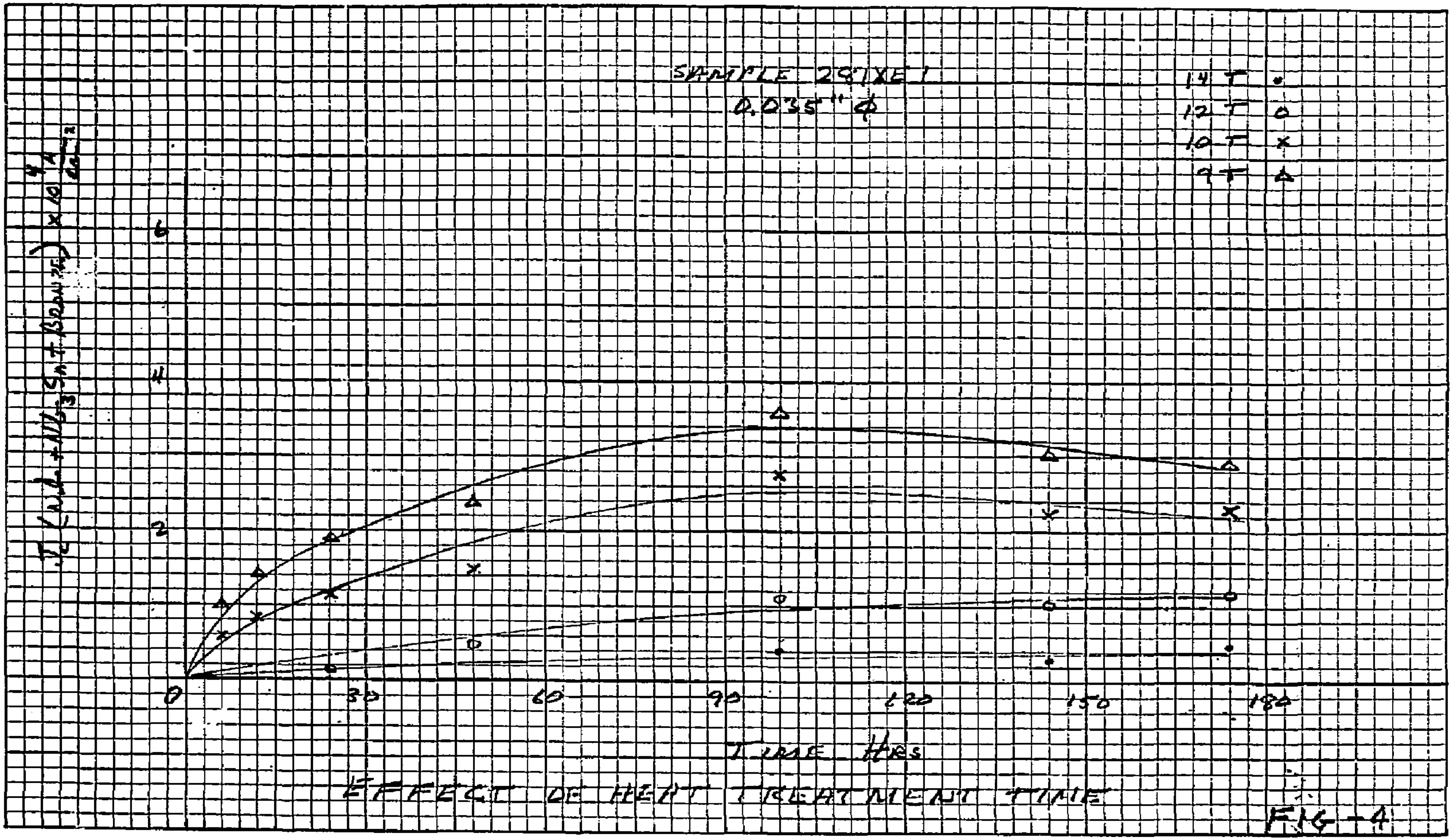
SAMPLE 29XE1
0.035" φ
14 T •
12 T o
10 T x
9 T Δ
6
4
2
0
30
60
90
120
150
180
TIME HRS
EFFECT OF HEAT TREATMENT TIME

FIG-4