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Metallurgy and Ceramics

AEC Research and Development Report

A NONDESTRUCTIVE TEST FOR INTERGRANULAR CORROSION IN STAINLESS STEEL TUBING

by

R. C. Robinson

Instrument Development Division

March 1956

E. I. du Pont de Nemours & Co.
Explosives Department - Atomic Energy Division
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ABSTRACT

An eddy current instrument is described which indicates the relative degree of intergranular corrosion present in austenitic stainless steel tubes. The instrument is applicable to the testing of installed tubing. One per cent differences in resistivity were detected between sample tubes and standard tubes of stainless steel. A sample judged to have very slight intergranular corrosion showed a 14 per cent increase in resistance while a severely corroded sample showed a 286 per cent increase.

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A NONDESTRUCTIVE TEST FOR INTERGRANULAR CORROSION IN STAINLESS STEEL TUBING

INTRODUCTION

A number of short stainless steel tubes used in a plant application were found to lack mechanical strength. Metallurgical examination indicated that these tubes had been weakened by intergranular corrosion. This corrosion had been caused, prior to installation, by incorrect heat-treating and pickling procedures. Since it was not known how many of the tubes were structurally weak and since the tubes were welded into place and had to be kept in the system, a nondestructive test was needed to determine the extent of the problem.

SUMMARY

An instrument was developed that indicates the relative intergranular corrosion of austenitic stainless steel tubes by using eddy currents to determine their effective electrical resistivity. The instrument possesses good stability, and meter readings are unaffected by the electrical shunting of the installed tubing. Differences of one per cent in resistivity are detectable. The instrument was designed to test many short sections of tubing located in a congested assembly.

DISCUSSION

Austenitic steels are metallurgically unstable when heated in the temperature range of 425° to 875°C. Heating in this temperature range causes chromium carbides to precipitate in the grain boundaries. The metal then becomes subject to severe attack at the boundaries by even mildly corrosive liquids. This attack is referred to as intergranular corrosion[1]. In severe cases, the steel disintegrates into separate grains with an average diameter of 0.05 to 0.15 mm.

Several tubes of Type 304 stainless steel that had been sensitized by heating in the 425° to 875°C range followed by pickling in a solution of 12 per cent HNO_3 and 2 per cent HF failed in service. An instrument was needed to determine the condition of those tubes that were still in use so that additional failures could be prevented.

Since intergranular corrosion consists of a network of fine cracks along the grain boundaries, it was reasonable to expect that the electrical resistance of a corroded tube would be different from that of a sound tube. Five samples of the tubing, which had an I.D. of 0.220 inch and an O.D. of 0.405 inch, possessing various degrees of intergranular corrosion were obtained, and the electrical resistance of each was measured with a Leeds and Northrup Kelvin Bridge. The results are shown below:

Sample Designation	Resistance of 5.5 cm of Tubing, ohm	Resistance Normalized to that of R2	Relative Metallographic Condition
R2	0.000915	1.00	Sound
R1	0.000910	0.995	Sound
K2	0.001045	1.14	Slight corrosion
K3	0.001490	1.63	Appreciable corrosion
K1	0.003520	3.86	Severe corrosion

The sample designated as K1 and shown in Figure 1 was prepared by pickling for eight hours in a solution of 12 per cent HNO_3 and 2 per cent HF at 49°C to 60°C. The piece designated as K3 was similarly pickled, but only for four hours. Sample K2, shown in Figure 2, was a part of a tube that had failed in service. The pieces marked R1 (Figure 3) and R2 were untreated and were considered to be sound. A correlation between the relative condition of the tubing and its resistance is clearly evident.

The use of a Kelvin Bridge for field testing was not practical since the tubing to be tested was in an electrically closed loop. The above results, however, suggested that eddy current techniques might be a feasible approach.

DESCRIPTION OF THE INSTRUMENT

The instrument is shown in Figure 4. Its sensing element is the coil of an oscillator circuit. When the coil, or probe, is placed upon a sample of tubing, eddy currents are induced in the sample at the expense of energy from the oscillator circuit. The induced eddy currents produce an opposing magnetic field that alters the impedance of the probe. The amplitude of the oscillation is a function of the electrical conductivity of the sample, if all samples are dimensionally alike and the probe-to-sample geometry is uniform.

The instrument is shown schematically in Figure 5. The probe coil is connected to the chassis by 11 feet of RG-59/U cable. As first designed, the probe was a coil wound on a U-shaped core of ferrite material. Preliminary tests of installed tubing revealed that the meter read off-scale in every case. This was found to be due to the electrically closed path from one end of the tube to the other. This path was provided by the metal supports of the installed tubing. A two-coil probe with opposing fields was subsequently designed so that there would be no net flow of current in this external path. The final probe consisted of two U-shaped coils connected in series. Each coil had 140 turns of No. 32 enameled wire wound on a core of "Ferramic Q" material made by the General Ceramics Corp. The coils were spaced 3/8 inch apart, and were oriented with the unlike poles in adjacent positions.

The tuned-plate, tuned-grid oscillator has a "no sample" frequency of about 127 kilocycles. The output of the oscillator is rectified by a 1N54A crystal detector oriented to obtain a negative voltage suitable for biasing the 6SJ7. To obtain the desired sensitivity, the circuit was aligned by tuning both primary and secondary of the plate transformer for minimum meter deflection with the probe on a sound sample of tubing.

When the plastic probe head of Figure 6 is placed on a piece of tubing, the Q of the grid tank is reduced. This reduction in Q decreases the amplitude of the oscillations, and decreases the rectified voltage across resistor R4 of Figure 3. The higher the resistivity of the tubing sample, the greater the reduction in Q and in the bias voltage across R4. With a sound sample, potentiometer R4 is normally set to result in a 6SJ7 tube current of 50 microamperes.

The probe housing, as shown in Figure 6 was designed to provide a reproducible probe-to-tubing geometry. In addition, the tuned-plate circuit of the oscillator was aligned to resonate at a slightly higher frequency than the grid circuit, to minimize signal variations due to any remaining variations in tubing-to-probe spacing. When the instrument was in use, the operator's attention was focused on the probe to make sure that it had properly engaged the suspect tube. Since the operator could not observe the meter face while watching the probe, a neon bulb was provided in the probe handle, as shown in Figure 7, to indicate when a corroded tube was encountered.

When the needle of the Sim-ply-trol meter reaches a preset limit, the neon bulb lights and stays on until the microswitch in the probe handle is pressed.

Figure 8 shows the placement of parts and connections in the chassis box.

PERFORMANCE

The instrument possessed the required sensitivity to distinguish a very slightly corroded tube such as K2 from a sound tube such as R1 or R2. With the potentiometer R4 properly adjusted, sound tubes such as R1 and R2 read five divisions on the meter whose total range was 100, tube K2 read 50, and tubes K1 and K3 read off-scale due to their significantly greater degree of corrosion. A range switch, S2, permitted some reduction in sensitivity so that some of the tubes which read off-scale in the field could be compared with the slightly corroded tube K2. Even with this reduction in sensitivity, however, the severely corroded tube K1 still read off-scale.

The correlation of the meter readings with the relative degree of intergranular corrosion is shown below. The data were taken with an earlier, less sensitive, version of the instrument which gave on-scale readings with all tubes.

Sample Designation	Instrument Reading	Relative Metallographic Condition
R2	100	Sound
R1	100	Sound
K2	97	Slight Corrosion
K3	70	Appreciable Corrosion
K1	10	Severe Corrosion

The frequency stability of the finished instrument was good. After a 30-minute warm-up period, only long-term drifts of 20-30 cps were detected.

The instrument has a short memory after the probe is removed from a tube, but this is not of sufficient duration to be of concern.

The instrument was sensitive to the presence of magnetic materials in the immediate vicinity of the tube being tested. It was found, however, that the probe could be brought within 0.5 inch of the ferromagnetic nut at the lower end of the installed tubes without adversely affecting the reading.

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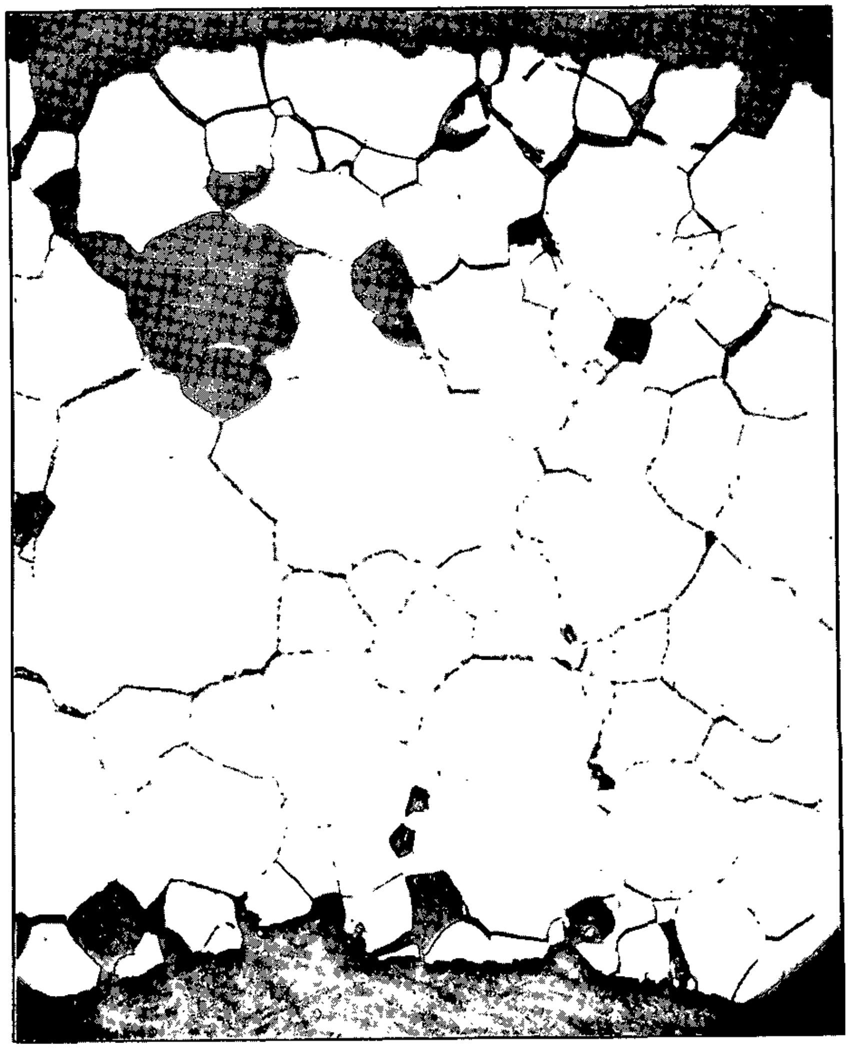
1. *Metals Handbook.* Cleveland: American Society for Metals, pp. 549-569 (1948).

FIGURE 1



MAG 60X

TUBING CROSS SECTION – SEVERE CORROSION

FIGURE 2

MAG 60X

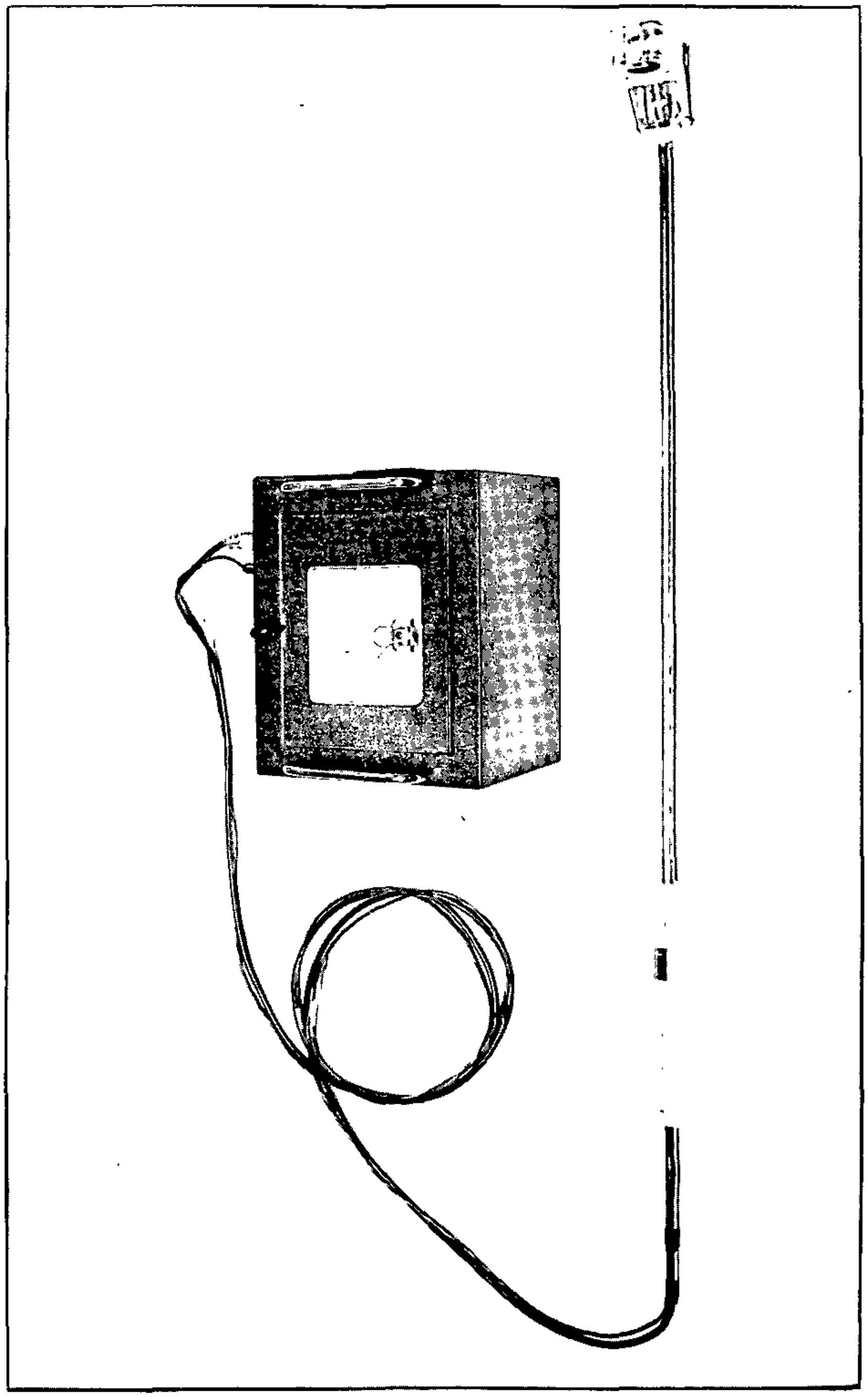
TUBING CROSS SECTION - SLIGHT CORROSION

FIGURE 3

MAG 60X

TUBING CROSS SECTION—NO CORROSION

FIGURE 4



TESTER ASSEMBLY

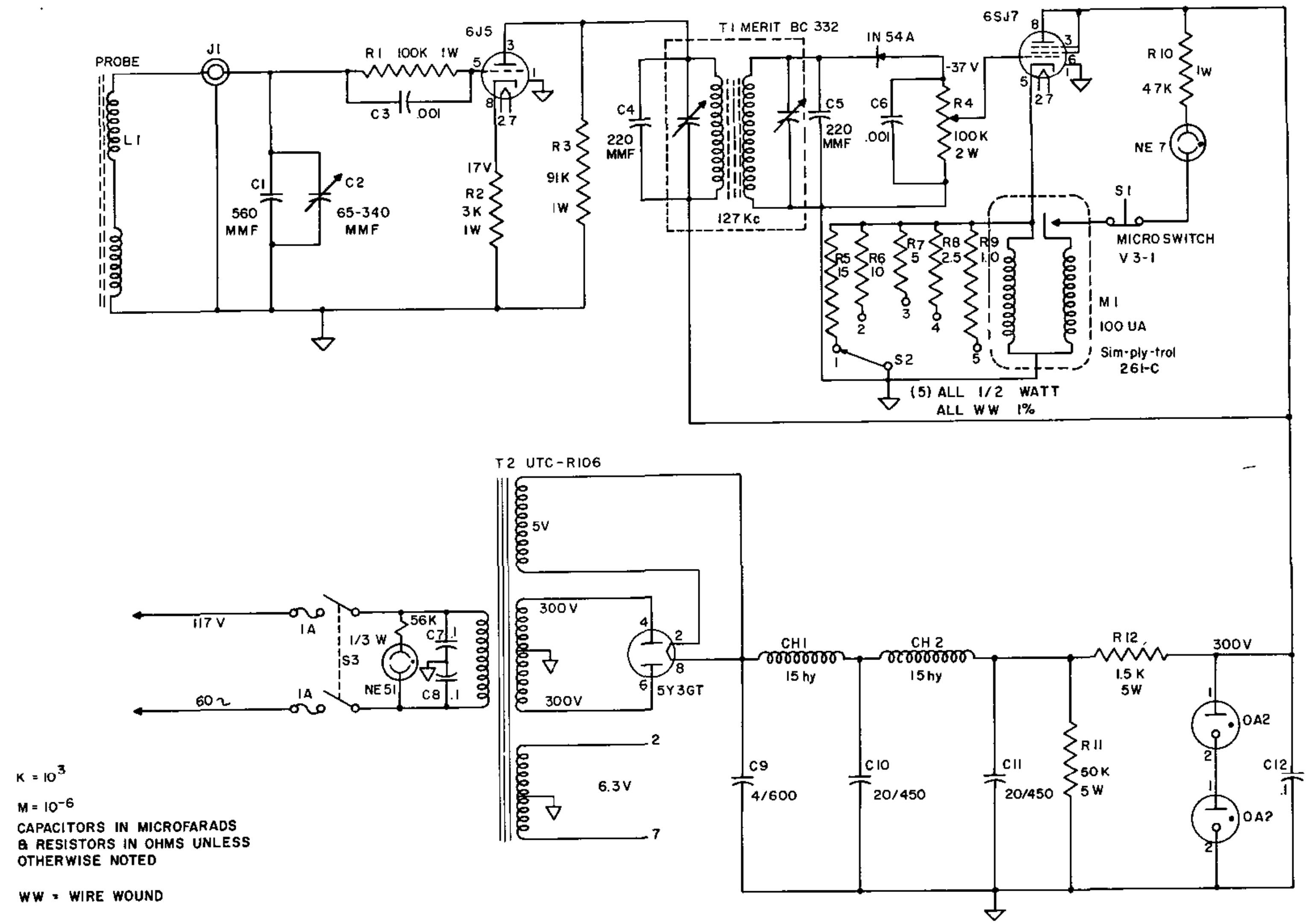
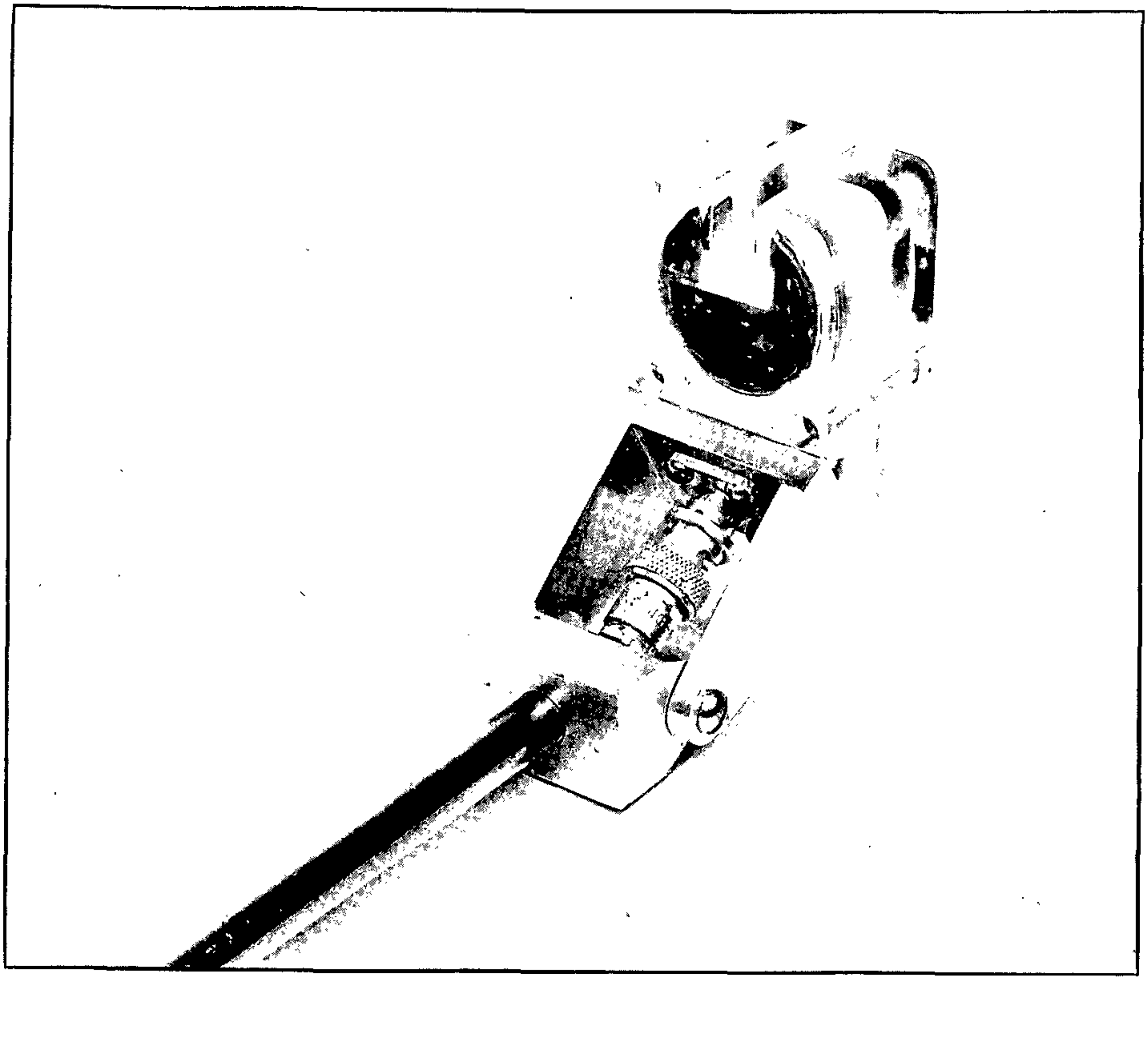


FIGURE 5

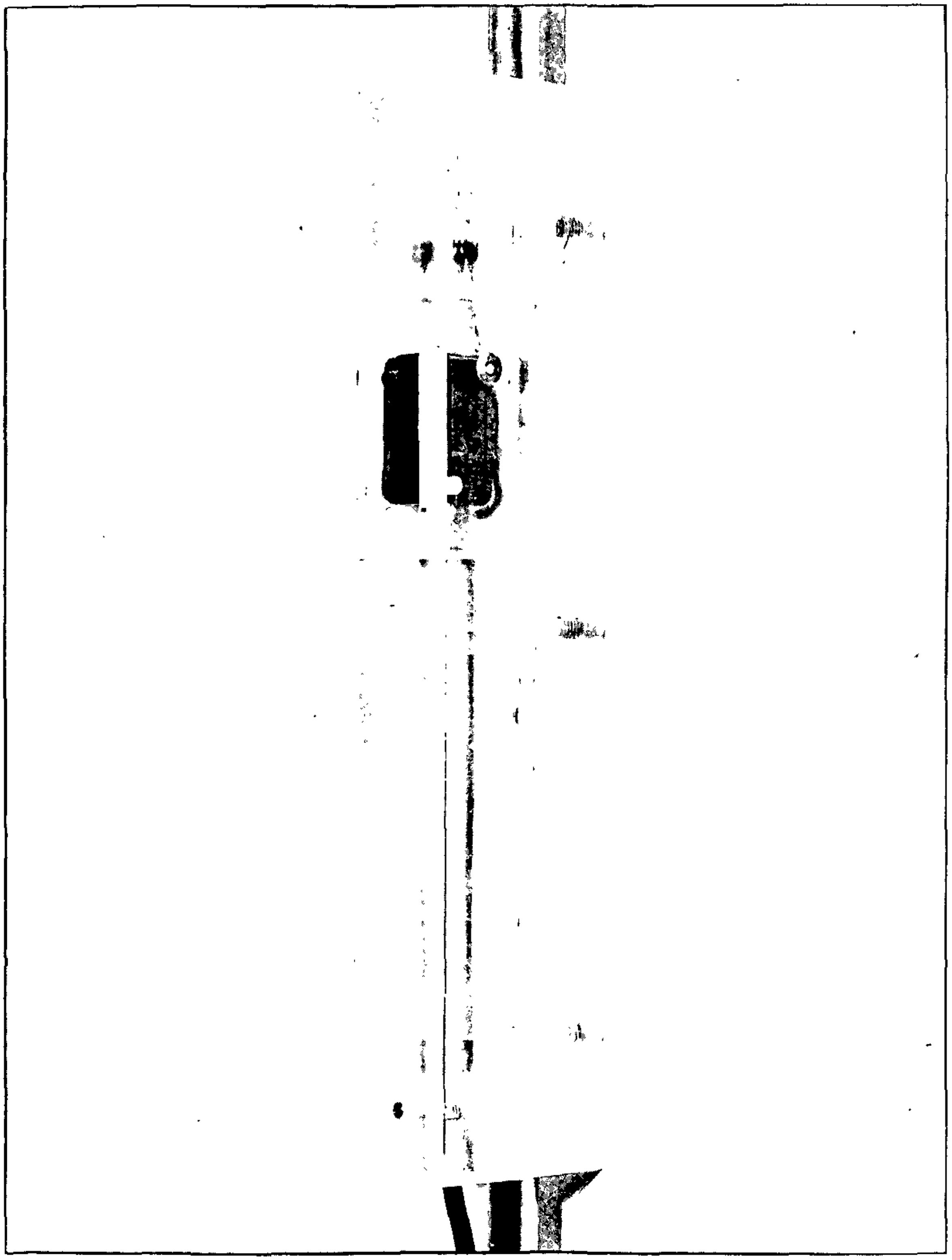
CIRCUIT DIAGRAM OF TESTER

FIGURE 6



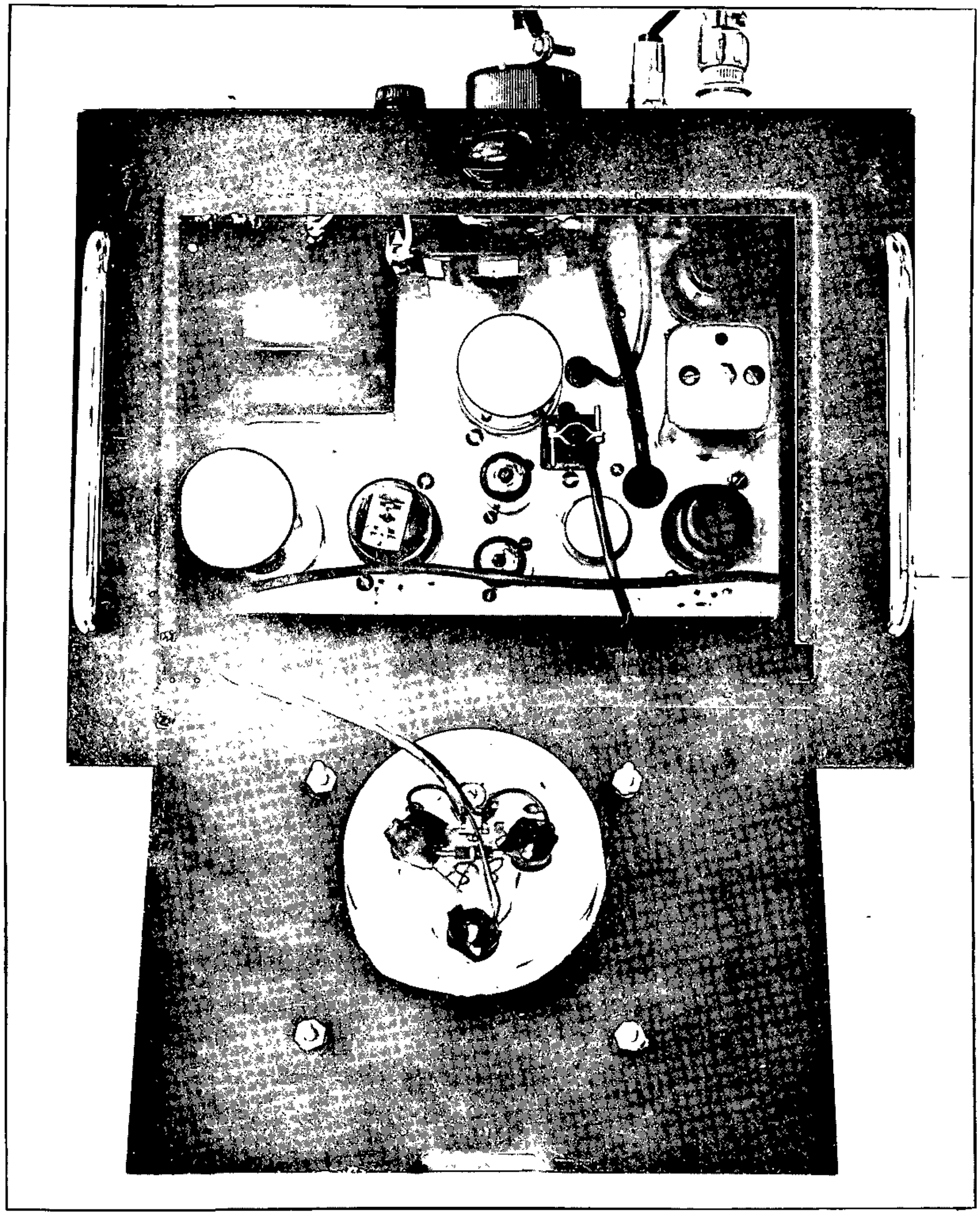
PLASTIC PROBE HEAD ASSEMBLY

FIGURE 7



HANDLE OF PROBE

FIGURE 8



CHASSIS ASSEMBLY