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XXXI. *On the Electrical Conductivity of Copper fused with Mica.* By Sub-Lieut. A. L. WILLIAMS, R.N., with Introduction by Prof. J. C. McLENNAN, F.R.S.*

[Plates V.-VII.]

INTRODUCTION.

WHILE acting as Scientific Adviser to the Admiralty, I had my attention drawn by Sub-Lieut. A. L. Williams, R.N., to some experiments made by him in the early part of 1919 at Cambridge, in which he found that samples of copper when fused with mica exhibited a remarkably large fall in resistance when gradually subjected to rising temperatures.

During a short furlough he was given an opportunity at the Admiralty Physical Laboratory, South Kensington, to develop this discovery and, on going back to duty, he left with me some notes embodying the results of his work. I have not had an opportunity of communicating with him again, but as the results are interesting it is thought they should be duly recorded. His experiments are described below, and accompanying them are some additional notes of results obtained at the University of Toronto by Miss Isabel Mackey and Miss I. Giles, who have followed up the subject still further.

J. C. McL.

A.

EXPERIMENTS BY SUB-LIEUT. A. L. WILLIAMS, R.N.

I. *Preparation.*

The samples for test were all prepared in the open on a piece of iron or copper plate—used as an anode—and a carbon rod as the cathode, the arc being struck at first between the plate and carbon, and then, when hot, to the mixture. The mica was first melted, then the copper added. In making up the samples studied, about equal proportions of copper and mica were used.

II. *Effect of Temperature.*

Resistance temperature measurements for two samples were made for a range of temperatures from 27° to 850° C. For sample A, the curves of which are attached, Graphs 1 and 2 (Pl. V.), the resistance fell from 16,000 ohms at 27° C., to 0·5 ohm at 850° C.

* Communicated by Prof. J. C. McLennan.

III. Notes.

(1) It was noted that the material was malleable at about 2000° C.

(2) A specimen piece was rolled at this temperature into a small rod 2.5 mm. in diameter for the purpose of ascertaining the specific resistance of the mixture. This was found to be as follows:—

25° C., Specific Resistance, 10,400 ohms.

30° C., „ „ 8,000 ohms.

(3) An attempt was made to obtain a sample of the mixture in the form of a very thin film for delicate temperature measurements, etc., and it was found possible to squeeze it out to about 1/1000 of an inch between platinum foil. It was not possible, however, to separate the film from the foil; but two pieces of foil cemented together by this fine film were found to be extremely sensitive to heat. They quickly responded to the action of infra-red rays from an arc about one yard away, notwithstanding the comparatively large volume of platinum to be heated first.

It is thought that with suitable films of the copper-mica mixtures enclosed in hydrogen it may be possible to use them for signalling purposes. It is also suggested that these films may be used instead of wires in microphones for sound-ranging, as the changes of resistance, due to changes of temperature, are quite considerable, being some thousands of ohms per degree centigrade with some samples.

(4) Attempts were made to make thin sheets by mixing the copper-mica material, finely powdered, with fine carbon, in the form of cane-sugar, and driving off the water by heating. The resistance of the resulting material was extremely high, but very regular thin sheets could be obtained in this way. It is possible, when the density of this mixture is increased by compression in an hydraulic press, that it may be obtained in sheets, rods, or other forms having a moderate resistance and yet possessing a high resistance-temperature coefficient.

5. Attempts to cast the material in various forms were not successful, partly owing to the difficulty in working with the requisite high temperatures. The material, when molten, is absorbed by such porous substances as porcelain, and if glazed porcelain is used the glazing melts and mixes with the material. It is possible that castings could be obtained by using fused quartz as a moulding material.

6. Attempts were made to make up similar compounds with the following metals and mica:—

Tin. The metal vaporized at too low a temperature.

Silver. Did not combine.

Platinum. Did not combine.

Iron. Combined, but no resistance temperature measurements were made.

B.

EXPERIMENTS BY MISS MACKEY.

I. *Experimental Arrangements.*

(a) The samples to be tested were all made in the open on a piece of iron plate used as an anode and a carbon rod as a cathode. The current was controlled by a large rheostat giving up to 30 amperes on the 110 D.C. circuit. An arc was struck between the plate and carbon and, when hot, the mica was melted and the other material added.

(b) A quartz tube closed at one end and covered with nichrome wire was used as a receptacle in which to melt the material and form it into a regular cylindrical shape for experimental work.

(c) A small electrical furnace was used to heat the material. It consisted of a circular porcelain foundation covered with wire and all was covered with asbestos except the two binding posts.

II. *Results.*

(a) *Mica and Copper.*—Mica and copper were fused on the iron plate into small lumps, and some of these were then finely ground into powder. No traces of mica or copper could be detected, only a uniform dull black powder. The powder was put into a quartz tube and heated, but this did not prove a satisfactory method of obtaining the mixture in the form of solid rods, as part of the mixture fused with the quartz, and it was found impossible to separate the two substances. When the quartz was broken, the copper-mica was found to be very brittle and not at all suitable for resistance measurements. Platinum wires were then fused into the ends of the copper-mica lumps which had not been powdered, and the variations in the resistances of these lumps were observed when they were raised to various temperatures.

Two different samples of copper and mica were tested in the furnace for variation in resistance with temperature (but only up to about $400^{\circ}\text{C}.$). In Case No. 1 (Graph No. 3), the resistance was found to vary from 4400 ohms to 300 ohms, while the temperature varied from $25^{\circ}\text{C}.$ to $400^{\circ}\text{C}.$ The specific gravity of the specimen was found to be 5.1, and as copper is given by 8.9, it will be seen that the specimen contained considerable mica.

GRAPH No. 3.

Temperature, $^{\circ}\text{C}.$	Resistance (Ohms).	Temperature, $^{\circ}\text{C}.$	Resistance (Ohms).
22	4,400	148	1,250
46	3,600	154	1,150
54	3,200	164	1,100
58.5	2,940	206	780
61	2,900	260	611
65.5	2,800	275	450
70	2,460	315	400
72	2,400	344	340
93	1,950	364	281
127	1,550		

In Case No. 2 (Graph No. 4) the variation in resistance was from 95,000 ohms to 3000 ohms, while the temperature changed from $100^{\circ}\text{C}.$ to $400^{\circ}\text{C}.$ The specific gravity was found to be 4.3.

GRAPH No. 4.

Temperature, $^{\circ}\text{C}.$	Resistance (Ohms).	Temperature, $^{\circ}\text{C}.$	Resistance (Ohms).
139	45,550	228	11,370
178	23,330	211	13,585
236	11,645	208	14,570
262	9,120	200	16,738
314	6,000	192	19,090
345	4,590	172	23,445
364	3,790	165	26,230
378	3,605	155	29,670
395	3,280	147	34,050
333	4,200	139	40,505
323	4,740	130	44,350
313	5,490	122	51,340
290	6,330	118	57,110
278	7,030	113	64,070
267	7,790	108	74,033
256	8,610	103	81,740
245	9,410	99	91,010
232	10,790		

From these results it would appear that an increase in the mica-content of the mixture raises the resistance at ordinary

temperature and causes the fall in resistance with temperature to be much more rapid.

While more brittle than copper, the copper-mica is not as brittle as iron-mica compounds described below. The hardness is almost the same as that of glass. X-ray photographs showed the composition to be quite homogeneous. The mixture was black with a dull metallic lustre.

(b) *Iron and Mica*.—Two mixtures were made as in the case of the copper and mica, and the temperatures and resistances were measured as before. In Case No. 1 (Graph No. 5) the resistance fell from 1300 ohms to 100 ohms on being heated from 25° C. to nearly 300° C.

GRAPH NO. 5.

Temperature, ° C.	Resistance (Ohms).	Temperature, ° C.	Resistance (Ohms).
26	1,350	303	91
42	980	308	90
47.5	890	165	235
52.5	825	138	310
54	790	116	380
104	410	97	490
134	350	75	650
138	340	83	590
178	219	64	765
195	180	55	860
209	160	51	905
218	150	45	1,010
165	280	40	1,070
214	170	38	1,110
263	115	33.5	1,203
282	103	31	1,240
294	95		

In Case No. 2 (Graph No. 6) the resistance fell from 32,000 ohms on being heated from 160° C. to 380° C.

GRAPH NO. 6.

Temperature, ° C.	Resistance (Ohms).	Temperature, ° C.	Resistance (Ohms).
250	6,100	294	3,380
228	9,050	282	3,900
280	3,870	255	5,970
318	2,410	242	7,350
335	1,980	231	8,440
344	1,780	222	10,380
360	1,550	214	11,720
377	1,280	204	15,750
360	1,550	197	15,900
336	1,980	187	19,550
322	2,360	175	25,210
309	2,730	164	32,100

The hardness was above that of glass, and the material was much more brittle than copper-mica and had more metallic lustre. X-ray examinations showed the mixture to be homogeneous. The specific gravity in Case No. 1 was 3·7, and in Case No. 2 was 4. The specimens studied were quite irregular in shape, but from a rough examination of the sizes of the samples, it appeared that the sample which had the higher mica-content was the one which had the higher specific resistance.

(c) *Aluminium and Mica*.—No fusion was obtained between aluminium and mica. The two seemed to remain entirely separate.

(d) *Antimony and Mica*.—The antimony when heated gave off dense clouds of vapour, leaving nothing to fuse with the mica.

(e) *Bismuth and Mica*.—The same results were obtained as with antimony.

(f) *Cobalt and Mica*.—Cobalt and mica were fused on the iron plate in the same manner as the copper and mica. The cobalt-mica had a very dull black colour and was very brittle, but hard enough to scratch glass. Platinum wires were fused in the ends with difficulty, and the resistance at ordinary temperatures was very great. When heated red hot with a bunsen flame, a current of about '020 ampere was obtained, using the 110 circuit.

(g) *Nickel and Mica*.—When nickel and mica were fused, the substance produced was very similar to cobalt-mica. When it was heated red hot, a current of about '001 ampere was obtained, using the 110 circuit.

(h) *Manganese and Mica*.—Mica and manganese did not seem to mix at all. In one test, the manganese was found to form a complete shell around the mica, and in other cases an X-ray photograph showed the two to be quite separate.

(i) *Silicon and Copper*.—It did not seem at all easy, if indeed possible, to fuse copper and silicon. The two substances appeared to be quite separate after fusion.

(j) *Selenium and Copper*.—These fused quite readily and formed a dull black substance with very little or no lustre. The resistance was found at various temperatures and a graph, No. 8, drawn. The specific gravity was 6·6, and the hardness less than that of glass. With this mixture it will be seen that a discontinuity occurred in the resistance temperature measurements at about 150° C. The explanation of this result does not appear evident at present.

GRAPH No. 8.

Temperature, °C.	Resistance (Ohms).	Temperature, °C.	Resistance (Ohms).
370	·219	124	·261
335	·207	115	·200
315	·200	108	·176
298	·188	104	·172
260	·173	101	·169
247	·164	98	·169
240	·164	95	·168
230	·162	92	·163
221	·157	90	·164
204	·152	87	·164
190	·147	84	·163
180	·139	80	·165
173	·136	78	·167
167	·136	65	·176
160	·135	61	·176
154	·130	59	·177
136	·333	58	·176
130	·317	44	·185
127	·300		

(k) *Ferro-Silicon*.—A sample of commercial ferro-silicon was also investigated. It was found to be very brittle and difficult to grind up into regular form for examination. In studying a sample, leading wires of iron were used, as platinum fused readily at the junction when the ferro-silicon was raised to a high temperature. When a graph was drawn between temperatures as abscissæ and resistance as ordinates, the result was a straight line showing that the resistance varied directly as the temperature, just as in the case of ordinary pure metals. (See Graph No. 7.)

GRAPH No. 7.

Temperature, °C.	Resistance (Ohms relative).	Temperature, °C.	Resistance (Ohms relative).
280	·092	148	·078
259	·089	139	·077
246	·088	126	·076
226	·085	118	·075
214	·084	109	·074
202	·083	96	·073
185	·082	84	·071
176	·081	79	·070
165	·079	71	·070

C.

EXPERIMENTS BY MISS GILES.

In these experiments a micrographic study was made of the plane polished surfaces of the fused copper-mica mixtures referred to above. These were made both when the mixtures

were at room temperatures and when their temperature was gradually raised by means of an electric furnace. The object in view was to see whether the fused mixtures possessed any crystalline structure, and if they did whether the increased conductivity observed with them on raising their temperature could be connected in any way with observable modifications in their crystal structure.

I. *Preparation of Specimens.*

In preparing these specimens they were first of all filed off to an approximately flat surface. The surfaces were then ground on a carborundum wheel, and after that on several successive grades of aloxite of increasing fineness. The grades used were those commercially known as Nos. 90, 150, 220, and 3F respectively. The polishing was then started with optical alundum and finished with jewellers' rouge. The two coarsest grades of aloxite were used on a flat metal plate, while the finer grades and the optical alundum were used on fine even linen fabric stretched over a smooth glass plate. The rouge was used on a piece of soft, smooth broad-cloth stretched over a glass plate. The plates used were fastened on a horizontal revolving table rotated by a small electric motor. In some cases the surfaces were etched with nitric acid of various concentrations ranging from strengths of 10 per cent. to 25 per cent. and even to 50 per cent. Better results, however, were obtained by the use of ammonia in solution, with a specific gravity of about 0.93. With this solution the specimens were found to be uniformly etched by an attack of about one hour.

II. *Optical Equipment.*

The microscope used was one of the instruments especially designed by Bausch and Lomb for micrographic work. For normal illumination the type of illuminator used was the usual reflecting disk of thin cover glass. In this method the light was projected at right angles to the optical axis of the microscope, reflected from the cover glass along the optical axis of the system to the specimen, and then back through the microscope. For visual examination the source of light was a frosted electric light bulb, while for the photographic work a small carbon arc was used. The photographic plates used were rapid panchromatic, and the shorter wavelengths in the illuminating beam were cut out with a Wratten and Wainwright filter. Oblique, in place of normal illumination, was used in some cases.

III. *Results.*

When examined under the microscope different specimens were found to exhibit different appearances. Most samples appeared to be quite uniform in structure, while in some many little globules could be seen, which from their lustre appeared to be pure copper.

Specimens which possessed a high temperature coefficient were found both under high and low power magnification to show no change in structure, either by normal or oblique illumination, when heated to temperatures as high as 400° C.

Pl. VII. fig. 1 shows the appearance of a specimen at room temperatures with a magnification of 46. The resistance of this sample, which was 3200 ohms at 21° C., fell to 1600 when at 95° C. The structure of the specimen appeared very uniform, and no copper could be discerned in it judging by metallic lustre.

Pl. VII. fig. 2 shows the appearance of this specimen when etched with ammonia solution for an hour. As pure copper was found to require approximately about seven hours' exposure to ammonia to bring out its crystalline structure, the markings on the plate may be taken to indicate the boundaries between copper and mica or the constituents of the latter. The regularity of the markings would indicate that the copper and mica fused into an intimate and homogeneous mass.

A specimen, whose resistance at 100° C. was found to be 95,000 ohms and only 3000 ohms at 400° C., was polished and examined previous to etching it with ammonia, both with high-power and low-power magnification, and with oblique and direct illumination.

Pl. VII. fig. 3 shows its appearance when illuminated obliquely under a magnification of 46.

Pl. VII. figs. 4 & 5 show the same region when illuminated by normally reflected light under magnifications 46 and 205 respectively. The structure in this case, as will be seen, is quite different from that shown in Pl. VII. fig. 1.

With the sample illustrated by figs. 3, 4, and 5 there appeared to be a great many streaks of light and dark, bounded by straight lines running in all directions, while in other specimens there appeared to be nothing uniform in the shapes of the patches. The portions of the surface which are dark in Pl. VII. fig. 3 it will be seen are light in Pl. VII. fig. 4. In this specimen much detail was brought out with the low-power objective. It was therefore used among others with low magnification to study the effect of any increase in temperatures. A water-cell provided with

running water was placed between the specimen and the microscope objective, in order to cut off the heat from the objective, and the specimen was heated up to 400°C . No change could be discerned in the appearance of the etching.

Pl. VII. fig. 6 shows the appearance of a portion of the surface at a temperature of 350°C .

The specimen was then etched with the ammonia solution. Here, again, the surface was found to be marked by fine lines after an attack of about an hour, but no copper could be detected. It was heated again to 400°C . after etching, but no change in structure could be observed due to the rise in temperature.

IV. *Resistance-temperature coefficient of Glass.*

In studying these specimens one gained the impression that they possessed a number of the characteristics of glass. In most cases the specimens were very hard, and one could easily produce scratches on a glass plate with many of them. It is known, too, that many glasses when strongly heated become electrically conducting, and with a view of making a comparison between the behaviour of these specimens and that of a sample of glass, some measurements were made on the resistance of a rod of glass when its temperature was gradually raised.

In these experiments a rod of "Schmelzglas" about 8.0 cm. long and 5.0 mm. in diameter was used. Short platinum wires were attached. These were then joined in circuit with the mains of the 110 volt D.C. circuit, and the glass portion was placed within an electric furnace. As the temperature rose observations were made on the current which passed and on the fall of potential between the ends of the glass rod, contact being made with the circuit at the platinum junctions.

In these observations practically no current was found to pass through the glass until a temperature of about 300°C . was reached. Even then the current was only of the order of 10^{-7} ampere, which showed that the resistance of the glass rod at this temperature was very high, practically about 10^9 ohms.

From this result it would appear that the high resistance temperature coefficient possessed by the fused copper-mica mixtures is something specific, and it does not appear that the remarkable property they exhibit finds a direct parallel in the behaviour of glass.

The Physical Laboratory,
University of Toronto,
May 15th, 1920.

Graph N° 1.
COPPER - MICA
RESISTANCE-TEMPERATURE CURVE

Graph N° 2
COPPER - MICA
RESISTANCE-TEMPERATURE CURVE

Graph N° 3
COPPER AND MICA

Graph N° 4
COPPER AND MICA

Graph N^o 5
IRON AND MICA

Graph N^o 6
IRON AND MICA

Graph N^o 7
FERRO-SILICON

Graph N^o 8
COPPER AND SELENIUM

1

2

3

4

5

6