BULLETIN

THE INSTITUTE OF PHYSICS AND THE PHYSICAL SOCIETY

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Announcements

Nominations for Members of Council

In accordance with Article 71 of the Articles of Association of the Institute and Society, notice is hereby given that the Council has nominated the undermentioned persons to fill the vacancies mentioned which will occur on 30 September 1967.

These vacancies have been caused by the retirements from office of Dr. M. R. Gavin, as Vice-President for membership and education, Mr. A. E. De Barr and Dr. W. C. Marshall as Ordinary Members of Council.

The nominations for Officers are suggested to Council by the President's committee, consisting of the President and the two Immediate Past Presidents, and the Ordinary Members nominated are selected by ballot from a long list of suggestions made by Branch and Group committees and by individual members of the Council.

Attention is directed to the terms of Article 72 which reads as follows: "After the notice mentioned in the last preceding Article (**i.e. this notice**), any five Members (**i.e. Fellows, Associates or Graduates of the Institute or other Fellows of The Physical Society**) may nominate within two weeks of receipt by them respectively of such notice, but not later, any other duly qualified person as a candidate for election to any one or more of the said offices by depositing such nomination in writing at the office together with the written consent of the nominee to accept office if elected. No Member may nominate more than one candidate in any year for any one vacancy."

In the event of there being a contested election for any office, ballot papers will be circulated in due course to Members.

Vice-President

A. D. I. Nicol, B.Sc., Ph.D., A.Inst.P.
Secretary, School of Physical Sciences, University of Cambridge.

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(For re-election.)

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Chief Scientific Officer, Ministry of Defence, London.
(For re-election.)

Ordinary Members

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Professor of Theoretical Physics, University of Manchester.

C. A. Hogarth, B.Sc., Ph.D., F.Inst.P.
Professor of Physics and Head of Department, Brunel University, London.

For Vice-President:

Dr. A. D. I. Nicol is Secretary of the School of Physical Sciences in Cambridge and a Fellow of Fitzwilliam College.

He graduated in physics at Manchester University in 1943 and after a period with the Safety in Mines Research Board and the Manchester College of Technology he worked at the Cavendish Laboratory, Cambridge, where he obtained his Ph.D. in 1952. After some years as Senior Physicist with the B.S.A. Group Research Centre he became Secretary to the Department of Physics at the University of Cambridge and took up his new post quite recently.

Dr. Nicol became an Associate of the Institute of

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1966 Holweck Medal and Prize

The Holweck Medal and Prize is a joint award of the Société Française de Physique and our own body, which is awarded alternately to a French physicist and a British physicist. The award was instituted by the Société Française de Physique and the Physical Society at the end of the Second World War as a tribute to French scientists who had lost their lives during the war and the presentation of the award has always been made an opportunity for the French and British societies to come together.

The Medallist and Prize winner for 1966 was Professor R. Castaing of the University of Paris and the Faculty of Science at Orsay and since it celebrated his work on electron probe microanalysis the presentation was arranged during a conference on this subject held in February of this year.

The occasion was the dinner arranged in connection with the conference and Professor Castaing was able to receive his medal and prize at the hands of the President of the Institute and Society, Sir James Taylor. We were greatly honoured that his Excellency Monsieur Geoffroy de Courcel, the French Ambassador, was present at this celebration. We were very happy also to welcome the President of the Société Française de Physique, Professor A. Abragam, himself a Holweck prize winner, and his wife and also the Assistant Secretary General of the French Society, Monsieur Netter, and his wife.

In introducing Professor Castaing Sir James Taylor explained the origin of the award and commented upon Professor Castaing's excellent experimental work on electron probe microanalysis. After the presentation Professor Castaing described the problems that beset him along his path; we are very pleased to be able to publish his talk in this issue.

Photograph taken before the presentation showing (left to right) Sir James Taylor, Professor R. Castaing and Baron de Courcel.

The early vicissitudes of electron probe x-ray microanalysis

I do appreciate the great honour that The Institute of Physics and The Physical Society has bestowed upon me in awarding me the Holweck Prize for 1966. I was deeply moved when I learned from the Secretary General of the French Physical Society of a distinction which seemed to be much more than I truly merited. This emotion had scarcely begun to wear off, and I was gradually settling down to my new dignity, when I learned some time later the exact term the English language uses for this distinction. In France I was 'Lauréat du Prix Holweck'; in Great Britain I became the 'Holweck Prize Winner'. You see, on our side of the Channel this comforting name 'Lauréat' suggests academic success; it is associated with the laurel wreaths placed on the foreheads of hard-working pimply youths, who have distinguished themselves in intellectual competitions. But the word 'Winner' sounds far better; it could only be translated into French by 'vainqueur'; how could I resist the temptation to call to mind the sporting glory, how could I refrain from imagining exploits on the sports ground even if I only go to the stadium nowadays as a spectator in the stand? You will agree that such a feeling is particularly uplifting, especially for one who has reached a sufficiently respectable age to be asked to talk about his reminiscences. This is what I shall attempt to do now.

It is just twenty years—at the end of January 1947—since I 'took my first steps' at the French Office National d'Etudes et de Recherches Aéronautiques. At that time, this organization was in its very early stages and it had the most modern methods for recruiting young research engineers. The decision to accept me came only after a wide-ranging and very informal conversation, in which the candidate was expected, I suppose, to let his hidden aspirations slip out. On that occasion I was led to review my tastes in sport, as to whether I preferred individual or team-games. It was clear that the interviewer would draw a pointed conclusion as to my aptitude for team research. But not all the allusions had such an obvious purpose, and I remember when the conversation turned to the comparative merits of Tchaïkovsky's symphonies, I preferred to take refuge in prudent generalizations.

They finally placed their confidence in me, in spite of the vagueness of my musical knowledge, and after some months at the French Air Ministry I found myself installed with the whole of the Materials Department in a little research centre right out in the country, 30 miles from Paris, near the little village of Le Bouchet. My laboratory consisted basically of four walls and an ill-assorted collection of scientific apparatus; but we were awaiting the delivery of two electron microscopes, which at that time were a real luxury. However, these most fascinating instruments were not due to arrive at the laboratory until the beginning of the next year, and I spent the whole of 1947 getting up-to-date with the facts of experimental physics. Finding leaks took up the greater part of my time; this is a good school of patience for an experimenter, especially when the vacuum-tightness depends on such infuriating things as ground joints and vacuum grease. Of course, even in those unenlightened times, the use of rubber gaskets was common practice, but they had omitted to tell me this at the University, and the electron diffraction camera, the running of which had been entrusted to me, dated from the enthusiastic period which followed the work of Davisson and Germer. But I had several strings to my bow; was I not a native of French Gascony, rich in great culinary traditions? In a flash of inspiration I had the daring idea of fitting my instrument with that type of rubber ring that I had known since my childhood and which is commonly used for home-made bottled preserves. It was a brilliant success, and once I had done it, my camera was perfectly airtight, with the further advantage of looking as familiar as a tin of 'foie gras'.

About the same time I was formulating other rather ambitious plans, such as one for a slow electron diffraction camera whence the pattern would be recorded on an insulating plate and read off by a scanning electron beam. This was a fine plan, but of course a bit difficult to carry out with the technical means which were then at the disposal of my laboratory, and the preliminary report I wrote on this subject is still devoutly preserved in the files of O.N.E.R.A.

All in all, my activities at this time covered various fields and it would be difficult to pick out a common thread; nevertheless, it was during this year of 1947 that I took my most decisive step, because in July of that year I married Madame Castaing who, I am glad to say, is present on this occasion. Of one thing I am sure; without her support and encouragement I should never have been able to complete my Thesis.

It was only during the following year that I began to have a notion of what that Thesis could be about. Having at last received the two electron microscopes which I had been promised I had undertaken the study of precipitation in copper–aluminium alloys and in this way I had got to know Professor Guinier, who at this time had his laboratory at the C.N.A.M. and devoted part of his activity to the O.N.E.R.A. During one of the conversations, which related mostly to the aluminium–4% copper alloy, θ' and θ'' phases and to polygonization, Guinier asked me my opinion about the possibility of analysing point by point, at least in a qualitative way, a metal sample by bombarding it with electrons and detecting the characteristic x rays. I remember how with the self-assurance that comes from youth and inexperience, I replied straightaway that to my mind it was very easy to do and that it was surprising that no one had done it before. During the next two years I was to find out that there were in fact some slight difficulties.

Electron probes of some hundred ångström units in diameter had, after all, already been produced, notably by Hillier in the United States for his attempts to microanalyse thin samples by the characteristic energy losses of electrons. For detecting the x rays we would use counters; the counting rate might perhaps be low, but this was merely a question of patience.

We very quickly agreed on a detailed plan of work. On the principle that we would use electron probes of some hundred ångström units we had to expect beam currents which would not exceed a few ten-thousandths of a microampère. We then made some measurements with a conventional x-ray tube, curved quartz monochromator and counter. Knowing that the beam current in the x-ray tube was of a few milliampères, a cunning extrapolation led us to predict for our future analyses the uninspiring counting rate of a few pulses per minute. Of course we were being too pessimistic as we had not considered sufficiently the reluctance of a bent quartz crystal to reflect correctly the radiation from a broad source; the subsequent operations were to bring us a most agreeable surprise, but at that particular moment we concluded that we would have to fall back on non-dispersive methods such as balanced filters.

Moreover, a conventional microscope would obviously not be able to locate the point of analysis, since we were very hopeful of attaining resolving powers close to the supposed diameter of our probe, and thus far better than a tenth of a micrometre. Pressing on regardless, it would suffice to use scanning electron microscopy, which would allow us to obtain far better resolution—so the books dealing with this subject said. In short, the basic idea was to construct an instrument provided with non-dispersive spectrometry and with a scanning electron microscope for viewing the object, which would of course have the tremendous resolving power of a few hundred ångström units. On the other hand we could scarcely hope to obtain any kind of accuracy for quantitative analysis. In reality things were to be very different, and ever since that time I have been somewhat sceptical about the virtues of the planned research.

Obviously the first job was to obtain an electron probe of small enough diameter. I thought very naïvely that I would merely have to overcome a small number of quite definite aberrations, which were carefully classified in specialized books such as the excellent one by Zworykin and others entitled *Electron Optics and the Electron Microscope*, which had become my bible and source of inspiration. But these authors had perhaps insufficiently warned the reader of the secret weapons that an electron optical device is liable to use when it is prepared to fight for independence; nor had they said enough about the basic elements which in fact control the path of an electron beam: namely the dust particles it encounters on the edges of the apertures, and the grease layers which cover the metallic parts whose surface is supposed to be an equipotential. I had many opportunities to convince myself that the scattering of electrons by organic layers is a quite general phenomenon, very easy to obtain; I thus discovered the general characteristic of every electron beam, namely its aptitude, once it is left to its inner instincts, no matter to how small a degree, for turning into a splendid oscillator.

In short, after a few months' severe training, I had acquired the basic virtue of every experimenter who becomes involved in this fascinating field of electron optics, and I would have been able to hold my own with an experienced charwoman; but on the other hand I had taken a solemn vow: never to do any washing up out of my laboratory time.

By the beginning of 1949, I had succeeded in injecting into a probe of about one micrometre a current of a few thousandths of a microampère, and of this I was very proud. Sure enough there were papers in which people claimed to have produced probes of two hundred ångström units, but I was not far from thinking that they were out-and-out liars. Moreover, I had realized that I would have to give up the resolving power that I had first so cheerfully envisaged; I remember my despair when I became aware of the work of Thomson and Whiddington, from which it was obvious that a common fifty kilovolt electron would progress in my sample by a good fifteen micrometres before it would stop—of course not in a straight line, and Lenard, with his coefficients of absorption, had given me some comfort; but the spell was broken, for I realized that it would be in any case very difficult to go much below a micrometre. It was a big disappointment, because even at that time a resolving power of one micrometre was not commensurate with what could be expected of an apparatus which was so similar to an electron microscope. This disappointment must have shown a little in the paper which I presented in July 1949 at the First European Conference on Electron Microscopy in Delft, for one of the most distinguished scientists among those who took part at this conference had not failed to ask me the crucial question: "Do you mean that, in any case, and even if the electron-optical limitations could be overcome, the resolving power would still be limited to around one micrometre?" I might have quibbled a bit, especially if I had known about the work that Wittry, and Duncumb, were to do on this subject a few years later, but in the heat of the moment I could only admit that "unfortunately, that was exactly what I meant". "I see," he replied. And when, a little later, I met him once more in the hall, he complimented me on my work with great kindness and courtesy, concluding with the words: "It's a very nice method; it's a pity it won't be any use." I wasn't far from agreeing with him, but as we say in France, "If you have drawn the wine, you have to drink it", and I had no option but to carry on.

I have very happy memories of this Conference at Delft. It was the first time I had left France and my wife and I were both keyed up and somewhat apprehensive at the prospect of my having to appear in public. We were staying at a lovely old hotel, the Twee Steden, which unfortunately no longer exists, and we spent a week there gorging ourselves with strawberries and creâm, and with real coffee, all of which were very rare in France in the period just after the war. We had made friends with two young British couples, the Agars and the Revells, who had come to Delft to give a paper on their experiments of phase contrast in electron microscopy, and who had put up at our hotel. It was there that I learnt how pleasant a conversation can be between people who were born on opposite sides of the Channel, provided that one takes care to avoid such burning topics as Rugger and the Five Nations Tournament.

Incidentally I had taken excessive advantage of Mr. Agar by getting him to translate my paper into English, between nine o'clock and half past eleven in the evening. I had been told that I would do better to present my paper in English, and I had believed this in my naïvety, because at that time I was still unaware of the fact that all British people understand French perfectly, even if they don't admit it. And this is why the first paper on the microprobe, by Professor Guinier and myself, was in fact written in English by an English scientist, and read with a terrible Gascon accent in a lovely Dutch city.

Once this memorable conference was over, I just had to set to work again, for the whole thing was still to be accomplished; first I had to set up a suitable spectrometer for recording the x rays. Moreover higher authority had decided that almost all the services of O.N.E.R.A., and in particular the Materials Department, would be brought together in the building at Chatillon s/s Bagneux where they still stand to this day. It was not without some nostalgia that I prepared to leave the rustic surroundings of Le Bouchet, where we lived in a pleasant environment of permanent camping.

Of course there were some drawbacks; for instance the liquid air supply for the pumps posed a problem which would have to be resolved with the means at our disposal. I had finally come to an arrangement with my former laboratory, at the Ecole Normale Supérieure; they agreed to refill the four-gallon drum that I took with me in the bus which provided transport for the personnel between Paris and Le Bouchet. The drum was filled in the evening and I carried it back to my home, as a rule in a taxi, for the passengers of Paris buses were not very keen on sitting next to a container handled with extreme care, and from which unpleasant fumes were escaping. The liquid air spent the rest of the evening and the night in our flat. By the way, may I ease my conscience and make a certain confession? Remember that in the aftermath of the war food rationing was still imposed in France; I was still young and inconsiderate, and I hope you will forgive me if I reveal, after a silence of twenty years, that one evening, with the complicity of my wife, I misappropriated part of the liquid nitrogen which was intended for the development of the microprobe to make a home-made ice-cream (about 2 cubic centimetres of it—and not very nice, at that!).

Once we had settled down at Chatillon, everything was to be brisker. The instrument was soon adorned with a bent quartz spectrometer which made it possible to get on to the question of quantitative analysis. Meanwhile I had tried to make a theoretical calculation of the intensity of the characteristic lines, and I had noticed that the number of parameters would be considerably reduced if, instead of measuring, as was generally accepted, intensity ratios between different lines, one compared the intensities of the same line from the sample and from the pure element. To test this principle, the ideal thing was to analyse diffusion couples, where the pure elements and various intermetallic phases would be found together. But the apparatus was not equipped with a viewing microscope at this time, and my rudimentary sample holder did not allow me to measure the motion of the object accurately. I moved the sample haphazardly, and when a discontinuity of emission took place, I proceeded to an electrostatic scanning of the probe, to trace the diffusion curve near the phase boundary. All this would have been very well if my electron beam had been good enough to behave correctly during the whole experiment. Unfortunately the measurements were accompanied by an orgy of gun flashovers and recentrings of the filament; and the current carried by the probe, when it did not change abruptly, drifted with wily slowness. This was the time when I had my greatest fits of despondency, and when the apparatus was dealt the greatest number of kicks. But one day, as I was observing under the optical microscope a diffusion couple on which I had been working away for several hours, I noticed that all the phase boundaries were covered with myriads of contamination spots. I suddenly realized that the discontinuities of emission I had observed really corresponded to a change of composition. Before this I was beginning to believe that they only existed in my imagination, or in that of the instrument. This was a revelation. At the same time I realized that until then I had never really believed in electron probe microanalysis: henceforth I would believe in it.

From then on, everything was to go smoothly, though a few bad days were still in store for me. It was in this way that I had undertaken the correction of the astigmatism of my objective lens: during my holidays in the south west of France I had made some calculations which showed that such a correction could be checked by the shadow of a wire placed at the level of the caustic surface. I had spoken about it to Professor Grivet, adding that I thought plexiglass wires could be used for the experiment; the technique of making them had been described by someone who had used them, I recalled, for collecting fine droplets of fog. Grivet thereupon decided that nothing would be better than enrolling me for a paper on this subject at the First International Conference on Electron Microscopy, which was to be held in Paris in September 1950. He told me about it, incidentally, eight or ten days before the opening of the Conference. To my indignant protest he replied in a peremptory tone that it was the best way to make me work. He was right, but I still remember the sleepless nights I spent before thinking, two days before my paper was due, of metallizing with chromium my Plexiglass wires which until then would melt obstinately under the beam.

What more shall I speak of, except the wonder I was to feel a few months later after setting up the viewing microscope. Everything became easy, and the needle of the meter was moving miraculously when I brought a precipitate under the cross wires of the eyepiece. To my even greater joy, some of the precipitates became fluorescent under the beam; this somewhat monstrous apparatus was a splendid toy after all.

Then more austere times came: the calculation of absorption and fluorescence corrections, the writing of my Thesis, the sharing of my time between the University of Toulouse, where I had been given a lectureship, and the O.N.E.R.A. where we had undertaken the setting up of an improved model of the microprobe. I analysed samples for various metallurgists; I strongly suspect some of them of having misled me intentionally on the real composition of their alloys, with the obvious purpose of setting a trap for me.

The improved model of the microprobe came out in 1955; Cosslett and Duncumb were developing scanning analysis in 1956, and electron probe microanalysis thenceforth was to grow as you know. My laboratory was also growing, but in a far less spectacular way. I discovered the pleasure of training young research workers, and of noticing that some of them worked better than I did. But I was denied the enjoyment one feels in twiddling the knobs and overcoming the resistance of a reluctant instrument, except at longer and longer intervals.

Nowadays I still have the opportunity, between painful sessions of writing in my office, to have a short break in experimenting by myself on the apparatus of one of my students, under the pretext of showing him how to work it. I wish I could tell you more about it; but I dare not, for in this field there is an outstanding forerunner, and for more details I invite you to read again the famous pages where Jerome K. Jerome relates how Uncle Podger went about hanging up a picture.

R. CASTAING