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NATIONAL METALLURGICAL LABORATORY

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH

JAMSHEDPUR



No.NML/Symp-4/55

10th March, 1955.

Respected Doctor Sahib,

I am enclosing herewith a copy of your lecture on Graphite, which we had recorded. I request that you may please find sometime to correct it or alter it for publication. I am also enclosing copies of the questions asked there-on to which you may kindly send us your answers. We intend having the publication out by June this year. An early compliance of my request will enable us to do so.

With respectful regards,

Yours sincerely,

Rabindar Singh

(RABINDAR SINGH)

RS:km

Encl:7

Dr.K.S.Krishnan, F.R.S.,
Director,
National Physical Laboratory,
Hillside View,
New Delhi.

NATIONAL METALLURGICAL LABORATORY

Symposium

on

"RECENT TRENDS IN THE FIELD OF PRODUCTION,
PRACTICE AND RESEARCH ON REFRACTORIES
USED IN METAL INDUSTRY"

January 19, 20 & 21, 1955

DISCUSSION

Paper under discussion :.....Dr. K. S. Krishnan's lecture on
Graphite
.....

Name of the delegateMr. E. H. Bucknall.....

Representing
(Organisation's name)Director, N. M. L.

A short note on the points discussed

Dr. Krishnan referred to the unsuccess of a Californian Professor in making "synthetic" graphite single crystals. I wish to raise the opposite question - Can one really make randomly orientated polycrystalline graphite, and if so how? As it seems to me the inherently flaky nature of graphite and the essential dependence of this on the crystalline structure, it must be a matter of the greatest difficulty.

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.....By..Dr..Krishnan.....

Name of the delegateS.Viswanathan.....

Representing Tisco.
(Organisation's name)

A short note on the points discussed

Good performance of crucibles as regards resistance to burning requires graphite oriented in the plane of the potters wheel. As such it should be a simple of matter in the light of the properties of graphite. The question of disorientation put by Mr.Bucknall, I feel has no practical application as far as manufacture of crucibles although of statistical interest.

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Name of the delegateDr. G. V. L. N. Murty.....

RepresentingChief Chemist, Tisco.....
(Organisation's name)

A short note on the points discussed

The interesting properties of graphite are made use of better in the resistance heating vacuum fusion techniques in vogue in the Continent than the high frequency heating arrangements which are more elaborate.

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.....

Name of the delegateParvez Mehta.....

RepresentingTisco.....
(Organisation's name)

A short note on the points discussed

Pursuing the point about orientation in graphite where thermal shock resistance is required I believe that it would be necessary to have a disorientated graphite structure.

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Paper under discussion :.....Graphite.....

.....

Name of the delegateT.V.Prasad.....

Representing
(Organisation's name)N.M.L.....

A short note on the points discussed

Why there is observed a difference in the oxidation characteristics of the various graphites, Madagascar, Ceylon & Indian varieties, even for the same sieve sizes.

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..... By Dr. Krishnan

Name of the delegate T.W. Talwalkar

Representing
(Organisation's name) Tisco

A short note on the points discussed

Mentioned that in Tisco we have used graphite spiral for heating.

SPECIAL LECTURE

ON

GRAPHITE

By

Dr.K.S.Krishnan,F.R.S.

Graphite occupies, as you know the fourth column of the periodic table that is - right in the central column of the periodic table - the premier place and its peculiar properties are in a large measure due to its disposition in the periodic table. It has six electrons. Two of them are rather rigidly bound to the nucleus. We shall not concern ourselves with those two electrons because they do not take part in any of the ordinary chemical properties and we shall naturally restrict ourselves to the other 4 electrons which take part in chemical reactions. These four - spectroscopically we should expect to be all of them of one kind - that is what the Spectroscopist would call in the two S-shapes and the other two in the 2P shapes. Whatever orbit they may be in, it is not necessary to know precisely what these designation mean, but I merely want to emphasize that theoretically at first sight one would expect two of the electrons to be of the one kind and the other two of the other kind. But actually in chemical combinations - what ultimately decide the nature of the combinations is in what manner the energy can be minimised because ultimately that is the criterion for deciding whether a particular reaction does or does not take place - the two types are hybridised in such a way that there is no distinction between the four electrons. There are two ways in which they can participate in a chemical reaction. One is whether there is an equilateral triangle of 2 electrons together situated and the four chemical bond associated with 4 carbons that can be directed towards the four corners of a tetrahedion from the centre, i.e. the four points are directed towards the corners and that structure duly found in it. It is a typical aliphatic structure and the structure of diamond corresponds to that. There is another structure which is very different but which has an energy as low as the energy of

tetrahedral structure so that the other structure is also a stable one and we wish to pass from one to another. The other structure corresponding to three of the directed towards the corners of equilateral triangle is 120° , all the three lying in a plane - this accounts for only three of the electrons in carbon atom and the fourth electron is a kind of a vagabond electron. It does not know where to stay and its position depends upon the structure of the material. It is capable of wandering about. There is no tendency for that electron to be localized - that is- the structure which you will find for example with benzene - i.e. three of the bonds are in a plane. One of them corresponds to the binding with the hydrogen atom, the other two with the two neighbouring carbon atoms and the fourth electron is not freely localised. The structure of what we call an aromatic compound in general of which benzene is an example, is on this basis namely, carbon atoms in a plane at 120° to each other and the fourth electron not being directly accounted for. I shall deal about the fourth electron separately.

Graphite is a typical example of the aromatic structure of elementary carbon. You remember the old controversy regarding the structure of the benzene ring. Most of the controversies centered around locating the fourth bond and we have numerous models for example, the plane ring model, the hexagonal ring model, the puckered mix model, the prism model, the arm chair model and many others named after the structure. Actually, there are many more models that you can describe merely in terms of such models and it is no exaggeration, there are nearly as many models of the benzene ring as there have been prominent organic chemists. You have a Kekule model, you have an Armstrong model, you have a Leydenberg model and if I think over, at least I might give you 3 or 4 more giving you an idea the number of models you can have. All of them ultimately, if you think of it, depend upon localising the fourth electron bond. You now know why all of them have some degree of validity. Any one model does not explain all the facts and that is why you have so many different theories as each author was interested in explaining the phenomena

and chemical data that he had on hand and so he found a model that could explain them. Now we know that all this controversy arises because they were out to locate the electron bond which in reality is not localised at all. It is a kind of vagabond electron. It does not attach itself to any single atom of the benzene ring, and it is free to travel over the whole of the benzene ring. It is not localised, that is why these models have certain limitations in explaining all the observed data. When you come to naphthalene, the odd electron can wander not only over the benzene ring but over the 2 benzene rings so that it has a wider range of migration. From direct energy considerations, this is what could happen because larger the range of migration the lower would be the total energy of the system. The smaller the range or the more precise the location of the electron, the larger would be the uncertainty in its momentum. Therefore would be the large space which you have assigned to the electron for moving about the lower will of corresponding nature. One can also think in terms of wave mechanics namely, a location in short space correspondings to associated electron waves having very short wave lengths and a short wave length means a very high energy for the electron and so from all the various points of view they are really analytically and mathematically equivalent, they have made convenient points of view for use. They are all based on different systems of study of fundamentally physical laws. You can definitely conclude that the odd electron can migrate over the whole of the condensed range and it is in this manner it can minimise the energy of the system and that would be a stable configuration and that is what is actually happening in graphite, for example, which is the limiting case of the condensed benzene ring compounds. Where the ratio of carbon atoms to other atoms say, hydrogen, if it is hydro-carbon, you start with benzene, naphthalene and so on to an extent of different directions to corone, and next you will find the number of carbon atoms. The ratio of carbon atom to other atoms goes on increasing and will ultimately the other atoms get squeezed out, when the whole of the space is filled up by carbon atoms. It is more or less pure carbon what we called adsorption, which is unavoidable in graphite at the surface may be merely extending the

unavoidable presence of hydrogen or other atoms that still remain over when you pass to the limiting case of hydro-carbon with infinite number of carbon atoms over the benzene rings the other atoms get fewer and fewer and perhaps ultimately they get squeezed out. With this structure you get many interesting properties. First, this odd electron can move freely from atom to atom over the whole flake of carbon as most of you know graphite has a regular hexagonal structure, definitely in alternative direction. When one lower of graphite, you know graphite crystal has a flaky structure. Each flake of graphite has some such hexagonal structure in which atoms in the flake are extremely rigidly bound to one another. That is why a single sheet of graphite can be regarded as a molecule by itself. Next molecule is another single sheet which comes above it which is not directly above it is likely displaced on the centre between the two rings corresponding to the corner of one of the rings in the next ~~plane~~ plane. There is a third one comes exactly above the first one. All can occupy the different position again, you can have the other alternative of AD,AD. Those of the two layers represent or you can have three layers represented ABC, ABC both these occur in nature and some crystal occur both the time with this very same part of the crystal. Some parts of the crystal A,B represent the other part of the crystal, then you can have A,B,C represent. The properties of other type are very similar with physical properties only by determining electron diffraction methods or X-ray diffraction methods. One can find the difference, but two structures atom by atom in the plane to one another of the neighbour are naturally very strong. The distance between the two carbon atoms in diamond and therefore the binding of the carbon atom in diamond but the binding between the adjacent layers is fixed in the force that are involved, and so you can by putting the 2 extremes the atom in the plane are very tightly bound than the atoms in the diamond crystal. But the binding between the adjacent layers is extremely loose, so you get diamond, one of the hardest substance, and on the other hand graphite has a peculiar properties, but the two adjacent layers are very loosely

bound. You can sort out one layer from another very smoothly which is just like a pack of cards, each card is still a rigid structure between the other. From that you will find that graphite being a good lubricant, At one extent you can have the hardest substance like diamond and with the same element due to this peculiar structure, which is equally stable associated with this you can also have on one side a good di-electric with a high transparency and it is a good insulator and on the otherside you can have a dark material. Just like an electron, metal can migrate freely from one end of the metal to the other. One is the insulator and the other a fairly a good conductor. There are some limitations. I shall deal with the limitation later. On the other side transparent substance here is a dark substance. One has a reflection co-efficiency on the surface is not high in the case of diamond, but it all penetrates and it reflects back. It is an artificial manipulation of the angle on the other hand you can have a reflection from a polished surface above 60-70% just like the reflection from any metallic mirror. It has the metallic lustre and various other properties which normally one would associate with the metal diamond; a good transparent dielectric with this electron you are able to make perpendicular to the plane of graphite flake. You can make this electron to and fro. You know what is happening when a charged particle move out on equivalent to an element of electric current. In the presence of magnetic field you can swirl the electron round and round just as you do in cyclotron. The charged particle as they come out is just swing round and round and that is what you can do in the case of graphite. You can do into a small extent almost any substance bound to the nuclear of the atoms as they are bound to the nuclear, so they can move around and around, whereas here just because the electron is free to move with a very wide range over the whole of infinite range of the graphite. The big orbit you can produce and induce the magnetism, which is called Dia-magnetism, which enables the substance because of the small orbit.

It is extremely small in the order of .5% per diamond whereas here when you swing it round about 50 times that has produced in a substance like diamond. If in the case of flake graphite apply parallel to flake you can make this electron. We are not quite sure even now whether in the perpendicular direction the conductivity may be almost comparable with the low conductivity of the insulators. It may be as good as an insulator which all the normal insulators which we know. No means of separating out any small effect of the buckling, a little buckling, will give you components of the conductivity which will be larger than the conductivity. Some of the semi-conductor when I mentioned that you take a thick flake of graphite, you can electroplate the surface of graphite so that the electrode-zinc and if you have a electrode there, there you can get a current. Get it there. There is some technical difficulty, they are leaves of a pack of cards that the edge is so thin and each one of them you can't really electroplate the surface of graphite so as to get most remarkable experimental fact that the resistance between the electrodes is quite independent, whether you use the thick crystal or thin crystal. It is due to all these circumstances that instead of specific resistance remaining the same and independent of thickness or the actual resistance coming down rapidly depends upon the thickness. Its total resistance which remain the same because the current cannot penetrate into the medium because that will be the direction in which it will be confined to almost to a surface layer and the part of the crystal of graphite that actually conducts that is effective in conducting is just a thin surface layer and therefore whatever may be the parading between the two, the resistance remains practically the same and had a thick of thin crystal, the effective part is only to certain surface layers and remains independent of them, and the resistance that we measure is the effective resistance of that layer and not quite a whole crystal and that is why we get a remarkable observation you get some total resistance. The resistance in the plane seems almost independent. The thickness of the crystal when you get a thin crystal or you get the total resistance, you can associate with this layer structure you can

introduce between the two layers because the layers are tied up. Introduce other atoms what is called the swelling in graphite is really a result of such sandwiching the other atoms between the layers you can get nitride radicle, oxy-hydro radicle, carbon radicle you get on all sorts of each layer of graphite is an iron coupled with electron. Potassium ion, sodium ion and metallic ion of the electrons can be removed. They can take part in binding them to the atoms that are sandwiched between and you get a graphite compound of various types and they can follow certain stoichiometric relations. You can get a number of such atoms according to the chemical laws. If you have less of them some layers are unoccupied, some other layers are completely occupied. You get many interesting results associated with filling up one or more of these layers you can do in various steps. Suppose you can do that by putting it in one of the electrodes in an electrolytic arrangement and you can pull them out with similar you can do all sorts of uncanny things with the crystal of graphite. Graphite is such as a metal, I have dealt with that in great detail in my papers. I gave a lecture before the Institution of Metals, and also spoke about it in the Fuel Research Institute as one of the Foundation Lectures because graphite as a coal is carbon at various stages of carbonisations. One can deal with graphite as the limiting stage of conversion to carbon of the original organic materials. It is very interesting.

I shall deal with the refractory properties over the rest of the lecture, even there I shall confine myself to experiments which we have been doing in the Laboratory. I believe in giving a first hand account of things which we have also done because they are much more helpful than merely giving a report of work done by other people than I do. There is a well known method of determining the latent heat of evaporation of substances. The layer graphite evaporates and that is closely connected by a thermal law of the saturation vapour pressure, if it is too pure substance, the law is called Clausius Clapperon Equation. And it is a well known equation.

If you know the saturation vapour point at different known temperature you can calculate the latent heat of evaporation in substances. In order to do that Nuxergen originally developed a method, normally you can take a cavity in which the substances in equilibrium and it is filled with vapour, you puncture a small hole in a thin wall of cavity through which the vapour can effuse out. It is not flow in the ordinary sense, it is effusion in vacuum. If you know the rate of effusion you can even collect the total number, you can find the rate of loss of mass by weighing at intervals or you can even count the number if the particles can be counted which come out in a given small solid cone and you can calculate backwards from that number what would be the vapour pressure. When you deal with the vapour pressure on some of the metals the pressure is so low we cannot apply any of the ordinary methods, because it is what they do to put the metal in a refractory and to keep it in a high constant temperature metal sublime or evaporates and saturate vapour effuse out through the whole if you know the rate at which it is being effused over the two face solid angle calculating backward you know the vapour pressure it is a very convenient method for determining the vapour pressures of substances which had a very high melting point, many of the metals can be studied in that manner. We are using exactly the same method for determining what is called - work function for electron. We did it originally with graphite. It is very convenient. We have a thin wall type of graphite Acheson which can be worked out, turned a lay from a stout rod of graphite you can send a heavy current of electricity carbon rods, through the thin wall of graphite as convenient - i.e. from conductivity. It is not too much of a conductivity, its single crystal and if it is in the same plane naturally the cavity will be very high, the resistance is too low. But Acheson graphite is merely an aggregated crystal oriented at random. If they are oriented at random the conduction in which the new crystal will be in the plain but the contact between one crystallite and next crystallite is not electrically perfect

contacted and therefore there is a resistance at each point of the contact which consists not a single crystal but at random each oriented crystal can have quite an appreciable resistance. The conductivity is good enough because the resistance of right magnitude by suitably choosing thickness of wall of the tube you can raise the tube to any required temperature and you can control the temperature as you desired by merely regulating current through it. We generally send the current from a generator at a very low voltage. We can send about 400 amperes and so you can heat the tube to a very high temperature to 2000° - 2500°C , but actually we do not work at the high temperature because most of the constant temperature can be studied at temperature less than 1500 - 1700°C and so we do not have very high temperature, because there is a small technical inconvenience in work on the high temperature. We measure the temperature with the help of optical pyrometer. We have the tube in vacuum of the chamber really constructed and inverted into double valved thermos flask. It is transparent cylindrical thermos flask. The bottom is flatty ground at base plate and put underneath the high power fusion pump which can keep the vacuum of the order of 7 mill. mer. Two electrons are fixed to the based plate and you have the double valve chamber is a very convenient because one can sort out the electrodes above the wall and circulate ~~the~~ the water between the two walls just insert the nipple at the bottom through an Indian rubber tube which is inserted right at the bottom so that you can keep the temperature of the walls of the chambers nearly the room temperature whereas inside the temperature is 1500° - 2000°C . When you work even with graphite which has a very high melting point even with that when you work at high temperature there is a little sublimation though in quantity etc. is extremely small since it is strongly absorbed in the material, graphite is black. So you get a very thin coating deposit of vapor graphite condensing on the inner walls of the chamber and since we are making the temperature from the optical

pyrometer unless we can estimate of the absorbing coefficient of this from time to time and allow and measure of temperature whereas we are interested in precise measurement of quantity of the known temperature. Our temperatures are known to be correct to 2 or 3C and therefore we do not normally work at high temperatures. There is nothing to prevent us from working at high temperatures if periodically we clean the surface or we may make estimate of the optical measurement by subsidiary source of light and the light which is constant you measure the absorbed co-efficiency periodically. It is very good refractory and we have thin wall while we are making at thin walled flake of graphite, we fix them while a flake of graphite coming in, send a current and raise it to any required temperature and small holes involved we vet the electrodes out we can get the rate of effusion by Faraday cylinder. We can calculate backwards the vapour pressure. From the vapour pressure we can calculate the latent heat of evaporation of the electrons from graphite. It is just work function which they determine by other methods. There is one advantage here when you deal with, you are all familiar with black body of radiation, the radiation from the surface of any actual body is not the same black body radiation which has emissivity which differ from unity. It has a similarly observed co-efficiency of the emissivity which is not unique, therefore, you do not know what to do unless you know generally emissivity in great detail, you build a cavity, take a small apperture in the cavity and whatever may be in the nature of cavities, the emissivity of the walls of the cavity however different it may be from one unity the radiation that comes out from the hole in the cavity is ideal black body radiation, as long as the hole is too big radiation that comes out is too rapid to be replaced with the radiation that comes from the wall to fill up the cavity all that can be nicely arranged. We completely eliminate the facts of the surface in thermaonics. The most serious uncertainty is the effect of the growth surface. It may be reduced the emissivity by the factor by 100 of the actual value whereas by

this method we get rid of uncertainty of facts we get thermaonic constant which has a very small cavity. We quote inside with many metal which we want to study, we can quote by thermal evaporation or by electrolytic deposition. We have quoted all the elements of the first transition group starting from titanium to copper, you can quote them completely you can get the characteristic emission of the metals, whereas the old method of studying thermaonic one had to draw it out in the form of filament and one of the metal that can be so drawn, there are limited temperature due to elasticity that would not permit too many elements. We are able to get by this method most of the elements which can be deposited electrolytically can be studied using graphite as the refractory. There is another line which is very helpful from the study of the properties of the refractories. We know the spectral emissivity at a different temperature from the graphite surface which we utilize afterwards for measuring the temperature with the help of optical pyrometer and we also build to wind one single round of wire to any metal which we want to study for emissivity. You can wind the wire around at the single loop tightly at the centre in an extremely thin growth of flake. One knows the precision temperature from the temperature of the adjoining portion of the graphite surface and so we know the temperature also of the metal surface, with the optical pyrometer one can turn the temperature from the emissivity and one can use the filament in the form of thin filament or thin foil later on, one can measure the temperature with the optical pyrometer without any uncertainty. I mean to the same accuracy one can measure the temperature of a black body and we had the data for a large number of refractory materials which had a definite chemical structure of oxides or oxide, those which have known chemical structure or otherwise, such a precision measurements are not justified. There is another problem which may interest you when you are dealing with the refractories when you have a thin walled tube and you send a current to a considerable portion near

the centre as long as the tube is long and then the temperature drops down to the temperature of the ends at every point you get the generation of heat due to electric heating and you had a loss both the radiation from the surface loss due to the conduction ultimately through the ends of the tube. And so one can formulate the differential equation at the sametime the distribution of temperature even in the case of filament and not talk of a thin wall tube the problem does not seems to have been solved. There is some difficulty in solving the differential equation but one who is familiar with the experimental details has some idea of magnitude involved. We find that there are 2 solutions to differential equation, to particular solution to be able to combine in them in order to get a general solution one has to satisfy certain condictions. These conditions are satisfied only in a small region of the filament near the centre. At first sight it looks as though of passing of interest. We want to know the distance of temperature over the whole, some associated with experiments. We find that long before we get out of narrow region. One condition of combining of 2 particular solutions is satisfied where all the solutions increases exponentially, other decreases exponentially, before we move out of that region. One that is decreasing is almost negligible in comparison with the other increasing rapidly, so that when we can get out of the region we do not need any more to satisfy the condition for combining the two solutions, because one has already become insignificant and though mathematically if you give to that solution we will not be able to verify that it is the solution of the differential equation but actually in practice it is a complete general solution and you can get the distribution of temperature over the whole range of the tube. There are innumerable emperical formulas that are being used in the trade as Bell & Telephone Laboratories, General Electric and in many other places. These formulas all come out to specialize cases at particular solution, we have complete description with a

simple expression of the distance of the temperature over the whole range from the shortest length to large length. Whatever the length it is immaterial, one can have the complete description that facilitates now the calculation of the distribution of temperature and one can choose the length of the tube which will have a constant temperature over any desired length near the centre. In a series of 6 papers, 4 of which, I think, have already been published and 5th & 6th I have sent to the proceedings of the Royal Society for publication in A series. It is in accordance with the publication we have discussed in detail the distribution of the temperature which is of great inferior in studying the refractory properties because of thermal conductivity changes with temperature, electric conductivity changes with temperature, emissivity changes with temperature, both total and the spectral with data, all of them we can now come to a detailed description of the behaviour of any specimen of Acheson graphite or any other graphites that are used in a refractory under given condition. Thermaonic part as I said is also very interesting. There again, in series of 4 or 5 papers, 4 is already in the press, 5th will be appearing shortly. We are able to study the semi-conductor, you put them inside and you get exceptionally simple method of studying semi-conductors which formerly could not be studied by the ordinary method of the depositing on the filament; now put it inside because with that the chamber attains the equilibrium we are taking out of a small hole so we are studying the properties of the saturated vapour in the chamber and so it is an equilibrium and we are not disturbed by the vogories or idiosism of the surface which are very large in thermionic. All that is rendered possible because graphite is such a wonderful refractory. You can raise it to any temperature and the composition remains the same and there is no question of having to know the previous history of the start with the specimen just as you can raise the temperature as quickly as you like and bring back as quickly as possible, all that is rendered possible just because of these peculiar properties. We

have studied thin graphite Acheson rods for various properties and also we have done some of the highly refractory materials like Molybdenum, Tungsten and Platinum and others which are also refractories, they are not in the sense you know they are used in Metallurgy and all of them come out simply from the experiment. The fundamental basis for all that is graphite tubes which is a refractory with which we do most of the measurements. Since it is a part of the Symposium, I would like to treat this not as a lecture but a part of the symposium and I shall pleasantly welcome any question and discussion on the subject matter of this lecture.

Thank you.

MRK:
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