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SOME RESEARCH IN MATERIALS IN BIRMINGHAM
I. THE UNIVERSITY
II. INCO LTD.

BY

J.B. COHEN

9 January 1967

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SOME RESEARCH IN MATERIALS IN BIRMINGHAM
I. THE UNIVERSITY
II. INCO LTD.

Birmingham has been the womb of English nonconformity for the last 300 years. It is the "bull-ring" - a massive modern rotunda of shops and offices - and its surrounding skyscrapers. It is the numerous amateur acting groups - the surrounding quiet rolling hills and the racing horses and cricket fields. It is the growing suburbs that are gradually surrounding these. It is the bustling industrial city - with only one good hotel. It is the crowded early morning and late evening trains packed with businessmen from their company's offices two hours away in London and the pack of chauffeur-driven cars that whisk these visitors to and from a day's work in their plants or laboratories. It is the proximity to Stratford, and the attendant boredom associated with the repeated race to the Shakespeare Festival to entertain visitors. (Did they really come for business? ....)

It is the home of one of the first generation of Red Brick colleges, the University of Birmingham. And a second academic child is being suckled into maturity, the new University of Aston. It is a city rebuilding itself with fervor for the new technological age -- a bit late, but working at it.

In this report, two of the larger activities in physical metallurgy in this major city are described. INCO's labs and the University of Birmingham. Research in materials at Aston is only just beginning, and therefore is not covered in this report.

I. The University of Birmingham

The monstrous angular red brick appearance still dominates one's impression of this campus. It is a large university, and one would think its enrollment is twice the 6500 students who study there. There are many new buildings growing there, but for the most part they will not be noted for their beauty! There is a multilevel parking lot for faculty and staff (fee: £5 a year) topped with tennis courts, and a weird cylindrical sociology center. This last building is indeed grotesque! All the offices are around the outer wall, and as one stands in the hollow center (being sprayed by an over-zealous fountain) the impression is of a typical prison cell block. (For more details on this architectural achievement see ESN 20-7; p.100.)

A. X-Ray Studies in the Physics Department

In the Physics Department, Prof. A.J.C. Wilson is still unpacking from his move from Cardiff. His lab is a bit empty at the moment, although a new Picker powder diffractometer is on order, but he is still quite busy with matters of the International Crystallographic Union. He has just completed work on X-ray line broadening from Ni powders, comparing the size determined with the techniques of variance he has developed. His plans include work on samples that exhibit effects of strain and faulting, as well as particle-size broadening. He also plans to look into the effect of thermal diffuse scattering on studies of line broadening. The Physics Department emphasizes nuclear physics, and teaching of crystallography and diffraction starts in the MSc program. A large laboratory for experiments in this area, including many crystal models, is being set up in the new physics building. Wilson will move there shortly as it is nearly completed. Dr. Black is involved in studies of liquids, using a twin-tube GE Unit.

B. The New Mineral Engineering and Physical Metallurgy Buildings

Mineral Engineering and Physical Metallurgy occupy adjoining new buildings. At first sight they look more like rapidly built temporary quarters. Externally, the buildings are angular and much use was made of precast concrete; they are on concrete stilts. Both buildings are similar in form inside and out so I will describe in more detail only the Physical Metallurgy building. The main entrance is obscurely located on the side of the building away from the main road, in a muddy marsh facing the soccer fields. Obviously, this will be an area for future building, and for the moment this "front door" is locked and one enters through the loading bay. Inside, most of the floors are wooden, the walls are unplastered brick, relieved by a tasteful use of large tinted windows and dark wooden panelling. Offices are reasonable in size and nicely furnished in "Danish modern." The building itself is three floors, in each of two large cubes joined on an edge. All of the labs are on the outer cube faces, and some of the offices are also on these outer walls, but many are clustered around wells which run through the center of the cubes, and are open to the sky, so that these offices also have large windows with "outside" views. It is much like the interior design of the metallurgy building at Illinois Institute of Technology. As one walks around the building he feels very much as if he is in a maze. There is plenty of room for expansion.

For some reason the buildings (which were dedicated November 12) received an architectural award, probably because they were all up and in use within a year. They are attractive inside, ugly outside, so I can see no other reason.

There are large and small lecture rooms, and several small conference rooms. Study rooms for graduate students are included. There is much equipment here, although one must remember that in a new building like this, the British Government, through the Undergraduate Grants Committee, is quite generous on capital items. It will be a long time before funds for expensive items will be available again.* There are two large Instron tensile testers and two table-model units. One large room is filled with new optical microscopes. ESR equipment was being delivered during my visit. There are three electron microscopes - two AEI instruments and one Siemens unit. The X-ray equipment consists of two film units and two Siemens diffractometers with Philips electronic equipment and X-ray generators. A new Cambridge microprobe is being used.

There is a library, a reproduction center for reports, and a well-organized supply room.

C. The Materials Research Center

An attempt was made to initiate a materials science option in the Metallurgy Department, but this has never gotten off the ground. Instead, a "Materials Research Center" has been set up in which certain expensive equipment can be used by other departments for a fee (which may provide a fund for new equipment and replacement items). This is staffed and run by a separate group, not part of the teaching faculty.

D. The Mineral Engineering Department

The Mineral Engineering Department includes mining, extractive metallurgy and ceramics. The undergraduate training is the same for all until the last half of the third (and final) year, when a student may pursue more detailed studies in one of these three areas. This is the first year of the scheme. Dr. A.W. Nicol, who worked with Prof. R. Roy at Penn State for two years, is starting research on effects of hydrothermal treatments on reaction kinetics in borosilicate systems. Some work on diffusion of Fe_2O_3 into MgO has been started. This is about all the research activity in ceramics. There is no

* Well-equipped materials labs are cropping up throughout this part of the world, and it worries me a bit because I think not enough attention is being given to equipment and facilities in the funding programs in the US. I hope to expand on this some time in the future, when more general impressions of the whole scene and recommendations are appropriate.

professor yet in this ceramic "sub-group," and it is obviously only barely under way.

E. Metallurgy

(1) The Undergraduate Program

Industrial and Physical Metallurgy are combined in the first two years of the undergraduate program. In the third year, about two-thirds of the students opt for Physical Metallurgy. The number in this option is 20 this year, up from 10 last year. In the first half of this last year, students in Physical Metallurgy take a series of one- to three-week courses on techniques, and present a critique of some area to the staff. In the second half, they do a small research project.

In the graduate program, there are about 35 research students and 15 postdoctorate fellows, lecturers, etc. There is room for twice this number.

(2) The Staff and Research in Physical Metallurgy

Quite unusual for an English university, there are three professors in the department, Dr. G.V. Raynor, Dr. A.D. McQuillan and Dr. Rae Smallman. The Department seems to be run by a "committee" of these three. Beyond this quite unusual structure for this part of the world is the usual European construction of senior lecturers and lecturers who help each professor handle the day-to-day supervision of large research programs involving many PhD candidates. Of course, depending upon their own initiative, and the professor, a large share of the work originates with this part of the staff.

Raynor maintains some research effort, primarily on rare earth elements and their alloys, but he is a Dean now. McQuillan spent about two years designing and equipping the new building. He is starting work on electronic devices, produced by deposition. Smallman has apparently concentrated on building the research activity. He is, as he has always been, an active golfer, and a frank outspoken critic. He is immersed in helping to build the British Institute of Metals into a more research conscious organization with more activity by the younger members, and in particular in starting the new Journal of Materials Science to be put out by this Society. On top of all this he enjoys being with his students and seems to spend a great deal of time with them discussing research.

Smallman's main activities concern attempts to obtain more quantitative information on defects using the electron microscope. A very large effort had been devoted to employing the kinetics of climb of faulted dislocation loops to obtain information on the fault energies by using movies of the electron microscope images. The technique allows considerable accuracy, about ± 5 - 10 ergs/cm^2. Much of his work in this area was described in my earlier report on the Manchester electron microscopy conference (ONRL Conference Report C-20-66). Briefly, the most striking result to date is the measurement of the single- and double-layer fault energies. Smallman's group finds that the double-layer fault has only a slightly greater energy than a single-layer fault in an Al alloy. He is now pursuing this work in three areas. First, he intends to try to measure the annealing twin-boundary energy in this alloy. Second, he is going to attempt to measure fault energies as a function of temperature. (At the moment, he is trying to repeat T. Ericsson's measurements in the Co-Ni system.) Finally, he hopes to study coarsening, by filming the growth of particles, and is looking into the theories about this in more detail.

Mr. C. Richards has just completed a PhD thesis under Smallman and Dr. I.L. Dillamore on the effects of grain size and texture in rimming steel and polycrystalline Fe-3% Si (plus 0.03% C). By various anneals, grain size is varied with a constant texture, or grain size is held constant and texture varied.

Texture was found to have a marked effect on the ductile to brittle transition, below which fracture was entirely intergranular. With near random texture in the steel, twinning in compression (below the ductile-brittle transition) occurred at stresses higher than the tensile fracture stress. Thus, the twins found in brittle tensile specimens occur during or after fracture, not before. (Slip was found to precede fracture.)

With the same texture in all specimens, this group observes the critical stresses versus grain diameter shown in Fig. 1 for twinning, yield, and fracture. (The results

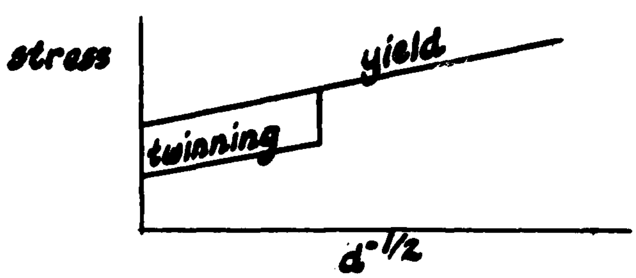

FIG. 1.

refer to the brittle condition when the two curves join.)

The results clearly indicate the role of stress concentration in slip bands; a definite length of slip path is required for each process.

Similar work is being done on this with the Fe-Si alloy, but it is just under way. In a purer Fe-Si alloy, an attempt is also being made to measure the velocity of twin growth over a range of temperatures, with particular attention to whether there is a change in growth velocity when twinning becomes less predominant. The technique being employed involves magnetizing a specimen and loading it. When twinning occurs, the change in sample shape causes a current in a search coil. (It may also be possible to measure fracture and slip velocities, as any change in shape produces a pulse.)

They have just found an interesting effect with pure iron. Magnetising appears to increase the twinning stress in a specimen.

Dillamore is one of the really "bright young lights" in this Department. He is particularly interested in textures, as evidenced by the work just described, and has a number of other projects involved with attempts to control texture for better properties.*

In rolled low carbon steel, his group has measured the dislocation density as a function of orientation (by tilting foils in the electron microscope to a given orientation) and also measured subgrain size and tilt (from the splitting of spots in the electron diffraction pattern). Calculation then yields the stored energy, shown in Fig. 2. Superimposed on this plot, is the relative frequency of orientations of grains in the plane of the sheet.

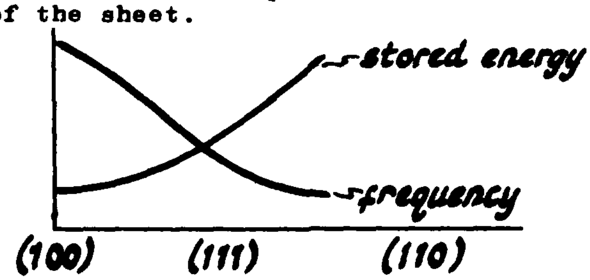

FIG. 2

* He took his PhD degree in the Industrial Metallurgy Group at Birmingham, and worked on textures then.

During recrystallization, grains near a (100) orientation tend to consume those near (110);and the resultant equilibrium orientation taking into account the frequency of each orientation originally present as well as the stored energy, peaks around the (111). However, the measured stored-energy is insufficient to drive a high-angle grain boundary except for those grains near (100) orientations. Orientations much beyond (100) must grow by a process of subgrain coalescence. If there is a second phase present, such as carbides or austentite, the approach to steady state is retarded, and distributions skewed toward the (110) orientation can be obtained. If the second phase is removed, say by decarburizing, the (larger) grains in (111) orientation grow quickly to consume the rest of the specimen and considerably greater (111) orientation can be achieved, providing twice the "drawability" and with an ASTM grain size of only 6, so that "orange peel" is not a problem.

Dillamore hopes now to improve low-carbon steel with respect to its magnetic properties. A flash intermediate anneal will be employed to initiate an intermediate texture. Recrystallizing in the $\alpha + \gamma$ range will be employed in order to utilize γ as a grain growth inhibitor. Subsequent decarburization and a final heat treatment, he feels, may provide a texture such that this material will be competitive with low grade Fe-Si alloys.

Preliminary studies are going on concerned with the effect of various recovery treatments on subsequent recrystallization textures.

A Siemens pole figure device is being used for the orientation studies, and construction of an automatic plotter will start soon.

Dr. R.W. Smith is working on the different effects of additions of Mg and Zn on the martensite reaction in Au-Cd alloys under a US Air Force contract. Explanations for the phase change in terms of ion-core overlap do not seem likely to him; the required contraction is much too large to be due to the cooling to M_s. A liquid helium cryostat for the diffractometers has just been completed, which will (through a bellows arrangement à la Prof. C.S. Barrett) allow deformation in situ. The M_d versus alloy content will be measured; perhaps a more basic pattern will emerge.

He is also interested in attempts to stabilize grey tin at high temperatures with Ge, following up some preliminary efforts of Prof. A. Ewald, Northwestern University. Since his training in Canada, he also has an interest in segregation in solidification.

Last year his group completed self-diffusion measurements and examinations of the Mossbauer spectra within 0.05°C of the melting point of Sn. They could find no evidence for an anomalous increase in vacancy concentration.

Dr. J.M. Pratt is continuing his studies of alloy thermodynamics, particularly of transition metals. Torsion-effusion, tin solution calorimetry and cells with solid electrolytes are all being employed.

Dr. C.J. Beevers is studying embrittlement in uranium and in Ni alloys, and crack propagation in Zr and its alloys (a USAF contract). Work is under way under his direction on deformation of two-phase systems (including cavities as a "second phase").

Under Raynor, Dr. I.R. Harris is studying the factors stabilising the structures of rare earth metals and alloys. These have a number of unit cells, cubic or multiple hexagonal close-packed cells. Much of the work involves magnetic studies to examine the population of the 4f state. He is just beginning some very interesting studies on the effects of high pressure (using a Bridgeman apparatus) and of deformation. Very novel effects have been discovered and will be worked on in more detail:

(1) deformation below the Curie point produces <u>less</u> faulting than deformation above. Magnetic interactions appear to stabilize the structure;

(2) deformation of rhombohedral samarium produces a double hexagonal structure, as if many extrinsic faults were produced;

(3) cerium exhibits extrinsic faulting in the <u>annealed</u> state, but deformation produces single-layer (intrinsic) faults. (Specimens are being quenched from various temperatures to see how the extrinsic fault concentration varies.) A similar phenomenon occurs in Au-In alloys near the phase boundary separating the fcc phase from a double hexagonal intermetallic. At the moment, only the asymmetry of the (111) peaks has been used to establish the presence of double faults. They need to look at the 200 peak, and peak shifts. Some electron microscopy is also under way;

(4) Pr and Nd (with double hexagonal structures) melt at about the same temperatures, but deformation broadens the X-ray lines of Nd, but does not do so to those from Pr. Both materials appear to be quite ductile.

(3) Industrial Metallurgy

The Industrial Metallurgy Department was "caught short" in the financial freeze and still occupies its old building. Research involves investigations on texture, rolling, drawing, and electrochemistry. Prof. J.B. Kushner, on leave of absence from Evansville (Indiana) University, is here, in the electrochemistry group, working on electron microscopy with the AEI instrument. He is studying the dislocation structure developed during the phase change that occurs when H_2 is charged into Pd. Research in this group also includes EM studies of the interface of battery plates. Some dabbling into more basic areas is under way in this same section. A home-built LEED unit is being used to study surface reactions and attendant changes in gas composition. Grain boundary motion is being examined in W, by pulsing a field on a specimen in a field emission microscope. In both areas, the group has just "gotten used" to the techniques.

II. International Nickel Co. Ltd., Research Laboratories

A. Organization

As many readers know, the International Nickel Company Ltd., England, and the one in the US are affiliates of the Canadian Corporation. R & D and market development on the British side are under Dr. G.L.J. Bailey, Assistant Managing Director of the English affiliate. Dr. W. Betteridge, well known for his work on high temperature materials, is Research Manager. From his office in London he supervises the main laboratory in Birmingham and a smaller one in Acton (London) which concentrates on precious metals. With a direct telephone link and only a two-hour train journey to Birmingham, there is a good deal of communication. (With an electrified rail line soon to be completed, travel time will be reduced to 1½ hours.)

At Birmingham, there are some 320 employees, about 65 of whom are professionals. Superintendent is Dr. R.A. Smith, who has been with the company for 15 years, since the completion of his PhD with Prof. Raynor. At Acton there are about 50 employees, 20 of whom are professionals.

B. The Buildings

The Birmingham group is housed in rather old buildings, which are, however, quite adequately equipped. There are two high-resolution electron microscopes, a microprobe, two X-ray diffraction units, and excellent and extensive facilities for mechanical tests and chemical analysis. There is considerable room for expansion, as many neighboring large buildings have been vacated by a production concern. Nonetheless, some thought is being given to moving to a country site, perhaps near London, to emulate the set-up of their US counterparts.

C. The Lab's Functions

The functions of the laboratories are perhaps best described by considering their mode of operation. The laboratory is broken down into the usual groups with group leaders, e.g., metal physics and special alloys under Dr. Ian Mitchell from Cambridge. In addition, Dr. D.W. Wakeman serves as a "free-lance idea man" and advisor to the Superintendent. As a result of customer usage or anticipated needs, or a potential market pointed out by the product or marketing groups in London, or as a result of their own thought in certain areas, projects are assigned to groups and individuals. (This follows committee discussion concerning all those involved.) These projects have time limits, and some "costing" is being attempted by asking the staff to indicate the percentage of their time spent on each of their projects. Each group can call on any of the others for aid or can offer assistance if they feel they can help. With a short write-up or so, a staff member can request permission to follow up any of his own schemes or offshoots of his projects, and can spend about a day per week on such items for a month or so. (By the time Smith gets to these, the work is usually done, so that, in fact, each of the staff can spend about 20% of his time on his own pet ideas!)

D. Research Activities

Some of the work concerns differences in needs in England and the US. For example, higher strengths are required in welds in building codes here than in the US and so the English affiliate cannot rely on work done in the US but must extend it. The laboratory now feels that the Nimonic alloys are well enough advanced not to require too much effort. Operating temperatures are essentially 1100°C, only 200°C below the melting point, and alterations in properties to suit particular needs can be readily handled. If sigma phase is troublesome in a particular case, alloy substitutes are available. A very large effort, however, is now being started on the problems of hot corrosion.

Some quite interesting and generally applicable principles of ternary phase diagrams have been used in designing protective coatings for Ir, Ru, Mo, and W. In alloys of each of these with Pd and Au, the tie lines in a two-phase field near the operating temperatures run from Mo, W, Ru or Ir, toward the Pd end of the diagram.

Because of this, coatings which do not melt at useful operating temperatures can be made for Mo, W, Ir or Ru from the tie-line alloy.

With Nb, the tie lines run toward the Au end of the diagram, and the equilibrium solution has too low a melting point to be useful. (The very same idea has been used at Rolls Royce in re-enforcing Nimonic alloys with W wire to greatly improve creep life. The system chosen was a Ni-Cr-W alloy which is in equilibrium with the W wire. The lack of interdiffusion was of prime importance in avoiding degradation of the fiber's surface.)

Some interesting studies have been made on Sn plate. By thinning through the iron substrate to the interface and using replication techniques, it has been found that the $FeSn_2$ compound that forms during the brightening (surface melting) of the Sn is in the form of a latticework, with the crystals in the shape of rectangular prisms. Considerable open pore space exists, although the compound does apparently reduce corrosion, because this is more rapid (through the pores in the Sn plate) without the brightening treatment. With a layer of Ni between the Sn and Fe, a continuous Ni_3Sn_4 layer forms during brightening, and corrosion resistance is improved by 50%. Six microns of Ni is sufficient, one is not. Intermediate thicknesses are being tested.

Ni-coated steels have been successfully prepared by coating steel sheet with Ni powder suspended in an adhesive and then rolling the sheet. The sheet is then ready for Cr plating. This procedure results in considerable savings over those involving multiple platings.

Another successful project which indicates the high level of understanding of Ni-base materials in the laboratory is the production of a ductile alloy for use with such acids as sulfuric. (This is a replacement for the very brittle Fe-Si alloys now in use.) It was found that Ni_3Si was not really brittle, as previously reported. The brittleness was really due to the fact that the compound formed slowly in a peritectoid reaction, involving a more brittle, higher silicon-containing compound. There was a considerable volume fraction of this brittle phase present after casting. Using the knowledge of the fact that Ti additions to Ni_3Al cause the reaction to change from a peritectoid to a peritectic, they made additions of Ti which accomplished the same thing for Ni_3Si. The compound had then sufficient ductility to allow 15-20%

elongation in a tensile test. Corrosion resistance was not good at intermediate concentrations of sulphuric acid, and the corrosion group suggested small additions of Cu and Mo which are known to be potent inhibitors when used together. (Why is not known.) The result is a new commercial alloy. Two-phase alloys of Ni_3Si with W, are under development, to improve strength.

Research is under way to improve ductility of Fe with lower Si contents, by adding Ni.

Attempts have been made to use the intermetallic NiAl as a heating element in the vicinity of 1400°C. Unfortunately, the oxidation products, alpha alumina and Ni-Al spinel, spall, and so far no way has been found to establish a coherent coating.

Studies are being made to improve magnetic properties by obtaining orientation in direct casting processes involving directional solidification.

In Nimonic alloys, Cr_7C_3 forms rather than the $Cr_{23}C_6$ found in steels. This forms more quickly on heat treatment than $Cr_{23}C_6$, but involves less Cr. The ductility minimum and peak in yield strength with increasing temperature appears to be associated with the time or temperature required for this intergranular precipitate to adopt a needle-like habit. The minimum can be eliminated with alloying additions, such as Ti, which tie up the carbon, reducing the amount of the Cr precipitate.

Finally, in powder products of Pt, evidence has been found that pores are effectively a second phase and lead to strengthening, although they are not so effective as precipitates in tangling dislocation or in inhibiting annealing. There is less dislocation content remaining with pores than in the presence of precipitates, i.e., the substructure is not stabilized by the pores as it is with, say, thoria.

One of the most pleasant features of this laboratory is the small (free) dining club for leaders of the small groups in the lab and executives from the London offices. Over an excellent meal and appropriate liquid refreshments, various levels of the organization get to know each other. How much more civilized than the usual noisy, crowded company cafeteria! Not being used to such fine treatment, however, it took some effort to return to discussions after lunch! (This is, of course, often the practice in this country, but it is dying out slowly for along with a modern building comes a noisy busy modern cafeteria.)

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13. ABSTRACT

Materials Research at Birmingham University is described with particular emphasis on the Physical Metallurgy Department, and its new building, organization and research. The activities of INCO Ltd's laboratory in Birmingham are also described.

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