The use of petrographic methods for the identification of non-metallic inclusions in steel.

(With Plates XIII to XVI.)

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Summary. It is shown that petrographic methods, using the microscope and micro-refractometer, can be a valuable aid in the identification of non-metallic inclusions in steel, even when their size is only a small fraction of a millimetre. Special ways of preparing the samples are given, and some of the methods used in examination are indicated. Natural chromite, corundum, merwinite, mullite, quartz, rhodonite, spinels, and zircon are taken as examples of the minerals found.

EVERY commercial steelmaking process yields a product that falls short of 100% purity. Though the skill of the steelmaker inevitably plays a large part in the control of quality, the nature of the impurities and, to a certain extent, the quantity present are related to the steelmaking process and to the type of steel that is being made.

Some writers on non-metallic inclusions have applied the term 'slag' to all solid impurities present in the ingot, but it is more usual to retain this word for the native inclusions resulting from reactions taking place in the molten metal, following additions to the melt. This entrapped slag represents a very small proportion of the total produced, the greater part rising to the surface and being separated from the metal. Native slag particles, together with foreign matter due in the main to chemical or physical attack on the refractories, constitute the non-metallic inclusion content of the steel.

It may appear from this that all non-metallic inclusions are artificial minerals, existing separately or in combination. This is not so. A number of natural minerals, for example quartz, zircon, and native chromite, are occasionally introduced into steel as a result of refractory failures. Even the minerals of slags, though artificial in origin, often have natural equivalents from which they may differ slightly in their optical con-

stants. Examples of such minerals are rhodonite and the iron olivine, fayalite. Mullite, though not a common mineral in nature, is of frequent occurrence as a reaction product in the foreign matter found in steels.

Previous work.

The examination of non-metallic inclusions has given rise to a considerable literature. The *Bibliography of Non-Metallic Inclusions in Iron and Steel*, published in 1935 by the Mining and Metallurgical Advisory Boards, Pittsburgh, Pennsylvania, contained over two thousand references. Though for the purposes of this bibliography the definition of a non-metallic inclusion embraced a much broader field than that included in the present paper, it is obviously impossible in a limited space to do justice to the large amount of research that has been done in this sphere. It must therefore suffice to refer to a small number of works that are readily accessible, and to rely on their bibliographies for further information.

From among the more important publications reference must first be made to the early classic by Benedicks and Löfquist (1930), which covers over three hundred pages. The series of five papers by Portevin and Castro, which appeared in the *Journal of the Iron and Steel Institute* between 1935 and 1937, is particularly noteworthy for its two hundred and thirty-five excellent photo-micrographs, taken with different types of incident illumination. Among more modern works may be mentioned the paper by Rait and Pinder (1946), entitled *The Origin and Constitution of Certain Non-Metallic Inclusions in Steel*, which combines the use of chemical, metallographic, and X-ray methods.

In view of the limited use that has been made of transmitted light in the identification of inclusions, particularly in this country, it is interesting to note that, as long ago as 1931, Hartmann employed an extraction method, followed by the mixing of the residue with Canada balsam, to make possible the use of a transmitted light petrological microscope for distinguishing between inclusions originating from the slag and from the refractory lining of the furnace; while in 1936 Yakshevich used a universal stage for the determination of the mineralogical constitution of some non-metallic inclusions in steel.

On the contrary, papers dealing with the mineralogical constitution of furnace slags are usually illustrated with photo-micrographs taken by transmitted light. Such is the case in Agrell's *Mineralogical Observations on Some Basic Open-Hearth Slags*, which appeared in 1945. The pioneer

paper by Whiteley and Hallimond (1919) on *The Acid Open Hearth and Slag* includes paired illustrations of samples as seen by transmitted and incident light—a practice that has much to commend it.

Methods used in the present investigation.

In the investigations described in the present paper, transmitted light petrographic methods have been mainly used, preceded by an examination in incident light—the metallographer's principal tool. Incident light should here be taken as including not merely incident illumination by coated glass plate or prism, but also dark ground illumination and the use of polarized light. Specialized methods of ore microscopy, calling for additional equipment and, in general, suited to larger objects than are available in many non-metallic inclusions, have not been used in the present investigation.

Where possible and advantageous, the normal microscopical examinations have been supplemented by refractive index measurements by the Becke method, in conjunction with the Leitz-Jelley micro-refractometer. The help of X-rays has been sought only rarely, and then mainly to support or contradict conclusions previously reached by petrographic analysis. The results considered in this paper may therefore be fairly taken as indicating what can be done in this sphere by anyone who has at his disposal a high-grade petrological microscope, well equipped for observations by incident and transmitted light, and possessing facilities for the taking of photo-micrographs, and either a comprehensive set of refractive index liquids or some form of micro-refractometer.

All of the transmitted light work has been done on chemically extracted inclusions.

Preparation of the specimens. The method of preparation of metallographic specimens for examination under the microscope by incident light is well known, though it may vary in detail from one laboratory to another. The surface to be examined is first carefully ground on a fine wheel until flat, the specimen being kept cool by frequently dipping it into cold water. The specimen is then polished on a graded series of emery papers attached to revolving tables, usually finishing on a '000' paper. The final polishing is done on lubricated rotating cloth-covered laps charged with 6μ and 1μ diamond pastes, though with the 6μ grade the uncoated surface of a disc of photographic paper is often preferred to cloth. In special cases a $0{\cdot}25\mu$ diamond lap is also used. The diamond

paste gives sharp outlines to any inclusions that may be present and avoids the building up of polishing medium round the harder particles and the dragging out of softer ones: it yields a clear polish ideally suited for observations of morphology, so far as this can be seen in section. Unfortunately it robs the inclusions of one of their characteristic features—differential hardness effects. Because of this it is often desirable to finish the sample with a light hand polish on a moistened Selvyt cloth, charged with γ-alumina, to produce a small amount of relief.

The method employed in this investigation in the preparation of thin sections through the extracted non-metallic inclusions was essentially that in general use for petrological specimens, modified by special precautions necessitated by the very small size of many of the samples. To facilitate the handling of inclusions, which may measure a small fraction of a millimetre in diameter, the method of mounting over a flake of muscovite recommended by Holmes (1921) for the preparation of slices through oolite-grains and organisms was adopted. Unless the inclusion was exceptionally large, 600 mesh carborundum only was used to rub a flat on it. This process was controlled by repeated observation with a low-power stereo-binocular microscope, viewing through the back of the slide with incident light being generally the more useful. A suitable flat having been prepared, cuts were made within the margin of the mica plate and the surrounding balsam was cut away. If this is done with transmitted light under a stereo-binocular microscope fitted with partially crossed polars, a satisfactory contrast can be arranged between the balsam and the mica; the limits of the latter may otherwise be difficult to see through the roughened upper surface of the mount. The mica was then cleaved and the tiny inclusion was lifted on its mount and stuck face downwards on a thin glass slide coated with Lakeside 70 plastic, prior to the rubbing down of the second side. Lakeside 70 was used instead of Canada balsam at this stage to reduce the risk of disintegration of delicate inclusions during capping.

In certain cases inclusions can with advantage be mounted without sectioning. This method is probably most suitable for those extracted from slabs, hot-rolled sheets and expanded tubes, in which inclusions consisting of glass spheres in the ingot have usually become deformed during hot work, finishing as approximations to triaxial ellipsoids. In such inclusions the length of the intermediate axis may be several times that of the shortest one. During mounting in hot balsam setting takes place with the shortest axis of the ellipsoid vertical, the resulting slide being a useful substitute for a parallel-sided slice.

Examination of the specimens. The methods used in examination are well known to mineralogists. They include observations of the degree of transparency, shape (including the crystallographic forms present), colour, cleavage, twinning, refractive index relative to the surrounding medium and, where possible, an accurate determination of at least the mean value. With the aid of polarised light it is determined whether the given mineral is isotropic or anisotropic, and, if it should prove to be anisotropic, the order of the birefringence; the maximum value of its extinction angle, which may be zero; whether it is uniaxial or biaxial, and whether its optical character is positive or negative. The determination of the optic axial angle of a biaxial mineral, or at least the value of 2E, may be well worth while, especially where it is impracticable to obtain a reliable figure for the mean refractive index. Even an approximate value, estimated from an examination of the curvatures of the isogyres, is better than none. The fact that this constant is influenced by solid solution concentrations makes it less reliable as an aid to identification in certain cases, but its determination may make it possible to place a mineral in its approximate position in a solid solution series. All of these optical properties, and the ways in which they are determined, have been dealt with in detail by Johannsen (1918), Hartshorne and Stuart (1960), and others, though some authors omit reference to the less commonly used methods. Determinative tables, based on various optical properties, are available in a number of textbooks, for example Larsen and Berman (1934), Winchell (1931) and, for slag and ceramic minerals, Rigby (1948), who also gives a list of references to photographs and a useful bibliography.

Examples from the present investigation. A residue that was extracted from a high chromium steel slab affords a good illustration of the triaxial ellipsoidal type of inclusion to which reference was made in the preceding paragraph.

A preliminary incident light survey of inclusions *in situ* in a polished section of this steel, followed by their examination in the rinsing water after extraction (pl. XIII A), indicated that they could be conveniently classified under three main heads, though mixed types were of frequent occurrence. The three primary types were: inclusions rich in lath-like crystals (pl. XIII B); inclusions rich in spinels (pl. XIII C); and glassy inclusions showing only surface globules (pl. XIII D).

Detailed examination of a number of inclusions of type 1 suggested, from the shape and optical properties of their characteristic phase, that they consisted of a merwinite-bearing slag. Thus the lath-like crystals

were found to be slightly shuttle-shaped, their birefringence was low, and their maximum extinction angle was approximately 36°. Additional evidence that these lath-like crystals were merwinite was provided by a conoscopic examination of the specially selected example illustrated in pl. XIII E. This gave a biaxial positive interference figure, showing the

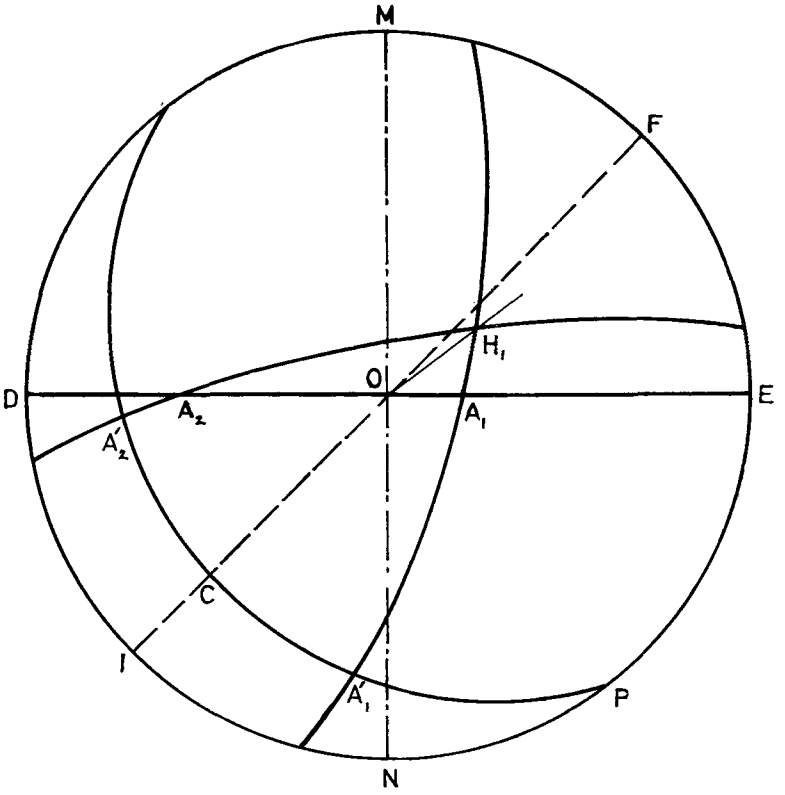

FIG. 1.

point of emergence of one optic axis. A determination of the optic axial angle was made by Wright's modification of Becke's method, for use when the point of emergence of only one optic axis appears in the field (Wright, 1911). Readings were taken on the isogyre, when straight and curved, with a modified Becke-Klein eye-piece, fitted with a calibrated coordinate micrometer scale, and plotting of the derived points was done over a 20 cm. Postel or angle meridian net. Since the optic axial angle was being checked for that of merwinite and direct measurement

of the mean refractive index was not possible in this case, the value obtained by Phemister (1942) for merwinite in blast furnace slag, namely $\beta = 1{\cdot}714$, was used. The final construction was as in fig. 1. The value found for the optic axial angle was $2V = 70° \pm 2°$, being the distance between A_1 and A_2 as read over the Postel net. This value is in close agreement with that found by Phemister (1942), namely $69° \pm 2°$.

Chromium-bearing spinels, probably agreeing most nearly in composition with picotite, are an important feature of these inclusions, and type 2 consists of these golden brown crystals in a glassy matrix (pl. XIII C). They rarely show a regular octahedral form, frequently being distorted to give elongated crystals with or without a dodecahedral face developed parallel to the flattened surface of the inclusion. When twinned they sometimes show complex geniculations (pl. XIII F).

Type 3 consists merely of a glass with globular sulphides in the surface (pl. XIII D). These show light grey under vertical illumination and are readily attacked by dilute sulphuric acid. Their identity was further confirmed by the orange ring test of Whiteley (Berglund: translated by Dearden, 1931). They were missing from inclusions extracted in hot hydrochloric acid, but retained when the iodine extraction method was used.

The use of exothermic compounds[1] to facilitate feeding during casting has sometimes led to the formation of foreign inclusions of considerable complexity. Since a wide departure from equilibrium conditions exists in these inclusions, the presence of incompatible phases is to be expected.

Small pieces of exothermic head, which may become entrapped near the top of an ingot, normally show an irregular, though somewhat softened, outline due to their origin as solid fragments; while original quartz, only partly changed to cristobalite and set in a porous and partially opaque ground mass, is a prominent feature, surrounded by phases mostly formed or modified by the exothermic reaction (pl. XIV A).

Of much greater variability and mineralogical interest are those in-

[1] Exothermic compounds, as used in the steel industry, consist of mixtures which react exothermally when strongly heated. They are made either as a powder, or as a refractory head—a reservoir, open top and bottom, with which a modified exothermic powder is incorporated during the process of manufacture. Though their precise composition varies with the maker, exothermic powders may be regarded as consisting of four intimately associated parts: a fuel (aluminium), an oxidizing agent (such as mill scale), a flux (such as calcite), and a refractory powder (often consisting largely of quartz sand). The object of using exothermic compounds is to retain a supply of liquid metal for a sufficient length of time to 'feed' or fill up potential contraction cavities, which would otherwise form in the main body of the solidifying casting or ingot.

clusions introduced by the incorrect use of exothermic powders. Since these are not designed to retain a fixed shape, much more evidence of fusion is to be expected. As is the case with fragments of exothermic head, original quartz from the sand in the mixture may be present, but perhaps the most characteristic phase in these inclusions is, in the author's experience, a pink corundum often heavily corroded or associated with the product of a peritectic reaction. Though it crystallizes in six-sided tablets, these are most often seen in section as pleochroic laths, giving straight extinction and showing maximum absorption when their length lies parallel to the plane of vibration of the lower nicol. These laths commonly show rhombohedral faces and twinning is not infrequent. Most attempts to obtain an interference figure in the obviously tabular crystals failed, partly because of this thinness, but also because they are rarely found free from obstruction. Clearly defined uniaxial negative interference figures, badly 'off centre', were given by a few of the laths that, by reason of their greater thickness, as measured in a direction perpendicular to the plane of the section, stood a better chance of spanning the depth of the slide. A fragment of one of these inclusions was crushed and a refractive index determination made by the immersion method, in conjunction with the Becke test. Though many of the fragments were still surrounded by the ground mass, a few edges were free and a value of approximately 1·767 was obtained (pl. XIV B). Sometimes the corundum laths have heavily corroded surfaces (pl. XIV C), but under favourable conditions these crystals may show very regular outlines, with a thin coating due to an arrested peritectic reaction. If this reaction, the product of which is apparently $CaO.6Al_2O_3$ (Filonenko, 1949, 1950), is enabled to continue by extremely slow cooling or reactivated by reheating to the peritectic temperature, the core gradually disappears and the thickened coating, sometimes lengthened by the addition of curved awns, may appear in section as a tube or simply as two parallel straight lines (pl. XIV E and F). These features are often seen more clearly by either vertical or dark field illumination in a solid polished specimen than by transmitted light in a thin section.

In striking contrast with the clearly defined crystals of corundum formed on cooling from the high temperatures of an exothermic reaction, are those found in inclusions produced when an alumina mould paint is washed by a limy slag. Irregular and spherical inclusions of this kind, as extracted from a casting and *in situ*, are shown in pl. XV A and B respectively. In those of irregular shape the proportion of mould paint

is very high, corundum being present as the original crushed fragments, though with their outlines considerably rounded. Their identity was confirmed by the observation of their characteristically low birefringence and by conoscopic examination, which showed them to be a uniaxial negative mineral. Though some of the globular inclusions consisted largely of slag or a slaggy reaction product, most were of intermediate types; one example, in which much original corundum still remains, is illustrated in pl. XV E and F.

Though mullite may be introduced into the microstructure of an inclusion as a result of peritectic reaction, this mineral occurs more usually, and often in considerable quantity, as a direct crystallization. Thus when a refractory of the fireclay type suffers a slag attack mullite becomes a prominent feature. A fragment of such a refractory, existing as a foreign inclusion in steel, was extracted and sectioned. It is shown between crossed nicols in pl. XVI A. Much mullite is present together with a cracked and partially absorbed grain of quartz. The optic axial angle, determined on the almost square cross-section of a similar sample of mullite, was found to be $43° \pm 2°$, a value agreeing with that given by Rigby (1948), namely 45°, but considerably higher than that given by Larsen and Berman (1934), namely 20° approx.

Rhodonite is frequently found in certain acid open hearth slags. It is the major constituent in the thin section of the slag inclusion shown in pl. XVI B. The dark phase present is an opaque spinel.

Reference has already been made to quartz, chromite, and zircon as examples of minerals of natural origin, which have found their way into steel by way of refractories. The quartz usually shows heavy cracking, due, at any rate in part, to passing through the α–β change. This sometimes happens several times, in alternate directions, during a series of heat treatments. Reaction borders are common. Conversion to a high temperature modification is not as common as might be expected. Chromite, showing a marked octahedral cleavage, is occasionally found as a cause of long, broken streaks on the surface of a cold rolled sheet (pl. XVI C, D, and E). An example of zircon with quartz, in a spherical inclusion found in a steel casting, is illustrated by Clark and Houseman (1959).

Conclusions.

It has been shown how some of the transmitted light methods of petrographic research can be a valuable aid to the identification of non-metallic inclusions chemically extracted from steel. There will be the

greatest gain when these methods supplement, not replace, those already in operation; but even when used alone they can provide valuable information some of which, such as the indication of the relationship between the phases present, is directly obtainable in no other way. Brief reference has been made to the origin of some of the inclusions described. To consider further the conclusions that should be drawn when a given mineral association is found to be present would be out of place in this context.

Acknowledgements. The author wishes to acknowledge his indebtedness to his colleagues R. Pemberton and J. W. Shillito respectively for the chemical extraction of the inclusions and for the making of the modified Becke-Klein eye-piece. His grateful thanks are also due to Professor A. Stuart, who supplied the standard muscovite used in the determination of the Mallard constant of the equipment, and to Mr. H. W. Kirkby, Director of Research, for permission to publish this paper.

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Explanation of Plates XIII to XVI.

Plate XIII

A. Residue extracted from a high chromium steel slab, photographed in water. Transmitted light, × 150 diams.

B. Type 1 inclusion *in situ*. Incident light, × 150 diams.

C. Type 2 inclusion *in situ*. Incident light, × 375 diams.

D. Type 3 inclusion *in situ*. Incident light, × 750 diams.

E. Extracted inclusion of merwinite-bearing slag. Transmitted light, × 750 diams.

F. Extracted inclusion containing chromium-bearing spinels (picotite ?), some of which show complex twinning, × 750 diams.

Plate XIV

A. Fragment of an exothermic head, entrapped near the top of a high-speed ingot. Photographed *in situ* by incident light, with partially crossed nicols in order to show the residual quartz grains. × 30 diams.

B. Fragment of an inclusion introduced by the incorrect use of an exothermic powder, immersed in a solution of refractive index 1·767. Note the optical merging of the lath with the fluid, as indicated by the arrow. Transmitted light, × 600 diams.

C. Inclusion introduced by the incorrect use of an exothermic powder. The heavily corroded plates consist of pink pleochroic corundum. The matrix is a glass throughout which small crystals of a spinel are scattered. The larger white patches are metal. Incident light, × 120 diams.

D. A thin section through the inclusion illustrated in C. Transmitted plane polarised light, × 120 diams.

E. An inclusion of similar origin, but from a different casting. Here the sharply defined corundum crystals are coated with a layer of $CaO.6Al_2O_3$, which preserves the regular outline while the peritectic reaction exhausts the core through rifts, which form at points farthest from the centre of the crystal. Incident light, × 300 diams.

F. A more advanced stage in the peritectic reaction. Two $CaO.6Al_2O_3$ shells, which are partially emptied, are approaching the appearance of hollow tubes seen in section. Others, still unperforated, remain with their content intact. Incident light, × 300 diams.

Plate XV

A. A cluster of inclusions, some irregular in shape while others are spheres, extracted from near the surface of a casting, made in a mould treated with an alumina paint. Incident light, × 60 diams.

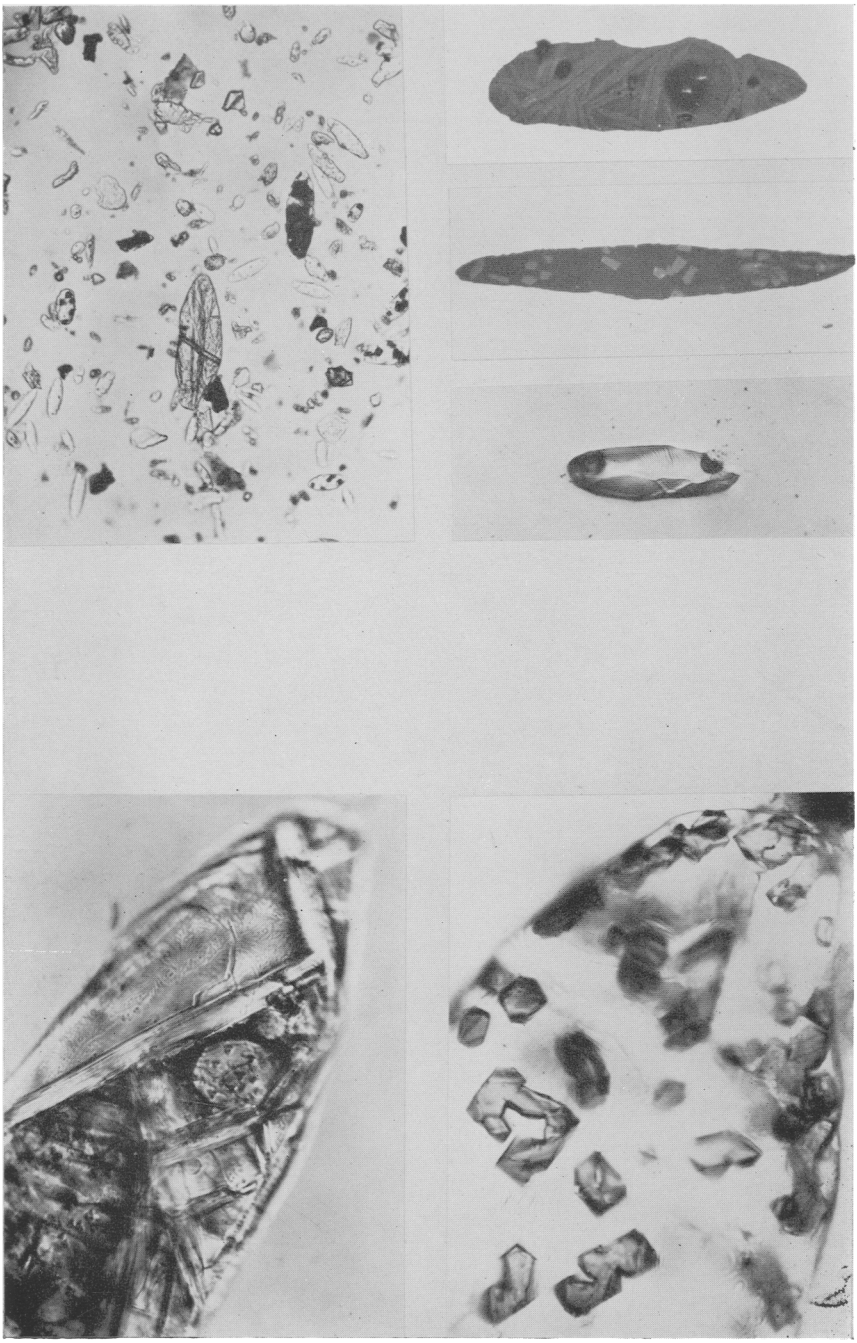
B. Section cut perpendicular to the surface of the casting, showing a mixed cluster of inclusions. Incident light, × 30 diams.

C. Thin section of one of the irregular shaped inclusions, which consists essentially of alumina mould paint. Transmitted light, × 60 diams.

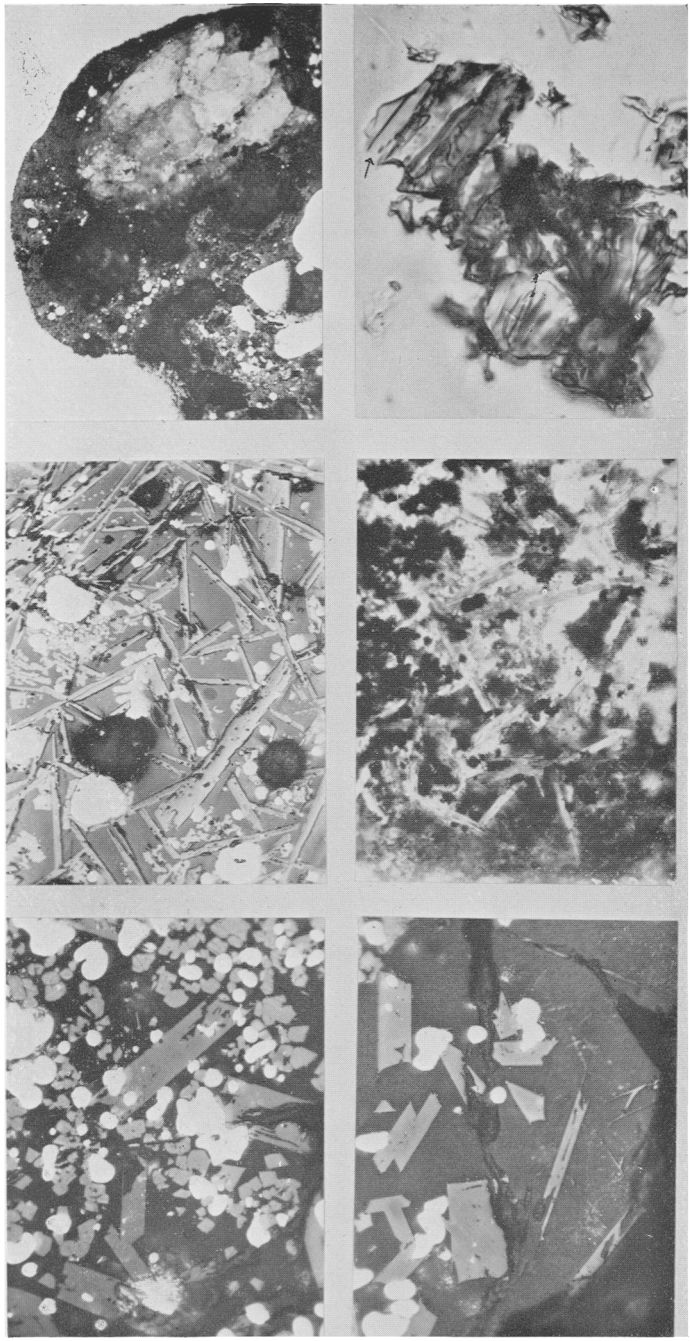
D. Part of the same inclusion showing the sub-angular shapes adopted by the particles of corundum. Transmitted light, × 300 diams.

E. Section through one of the spherical inclusions. Transmitted light, × 60 diams.

F. Part of the same inclusion, in which the corundum grains are bonded by a reaction product due to slag attack. Transmitted light, × 300 diams.



C. G. NICHOLSON: IDENTIFICATION OF INCLUSIONS IN STEEL



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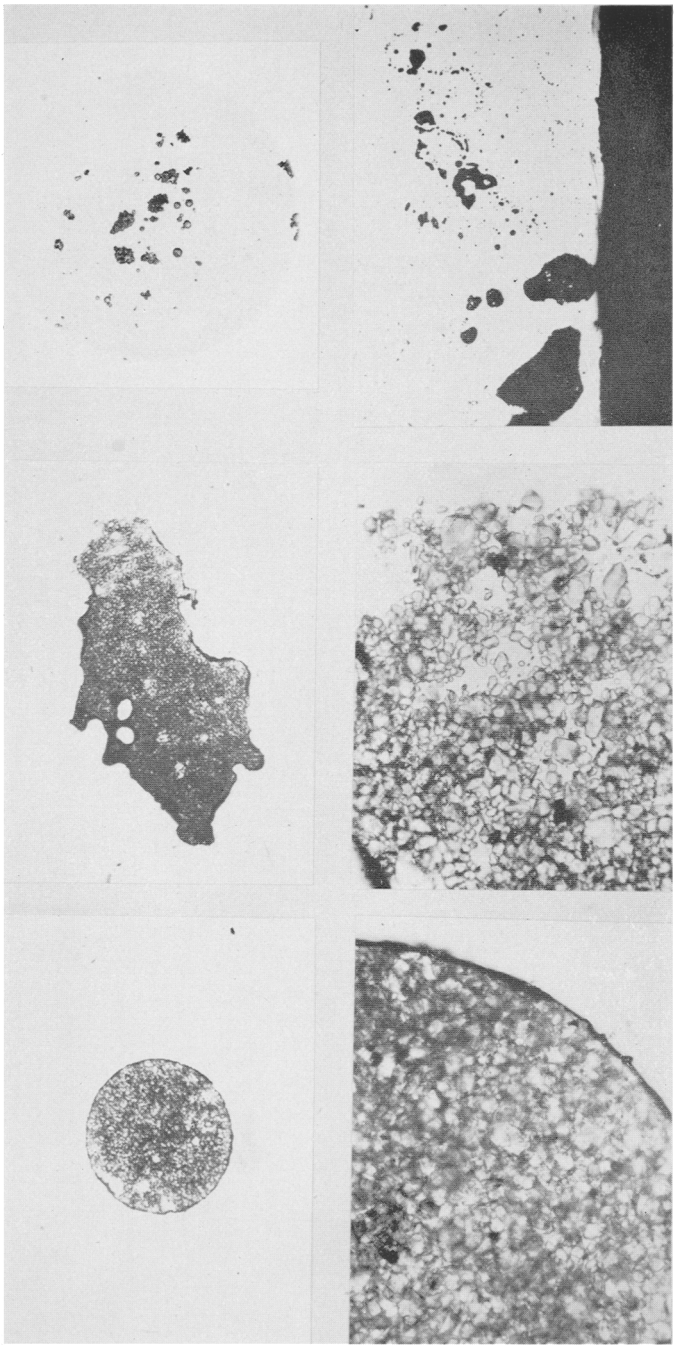

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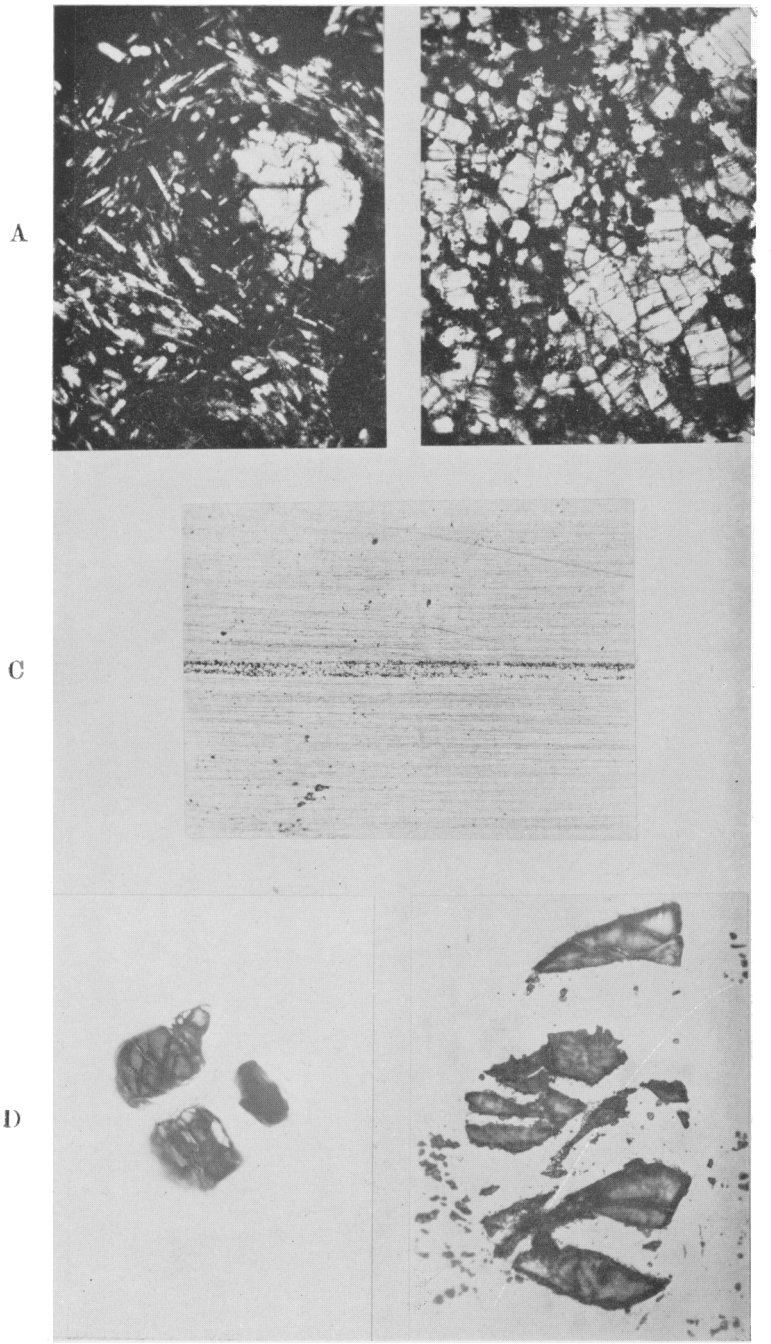

C. G. NICHOLSON: IDENTIFICATION OF INCLUSIONS IN STEEL

PLATE XVI

A. Quartz and mullite in a foreign inclusion introduced as a result of slag attack on a fire-clay type of refractory. The quartz, which is heavily cracked and corroded, is surrounded by a zone of glass. Beyond this relatively clear space, a dense crystallization of mullite is seen. Transmitted light with crossed nicols, × 120 diams.

B. Rhodonite, associated with an opaque spinel, in a slag inclusion extracted from a billet of acid open-hearth steel. Transmitted light, × 120 diams.

C. Streak on the surface of a pressing made from a cold rolled sheet of an 18/8 type of stainless steel. Incident light, × 6 diams.

D. Three fragments of chromite of refractory origin, extracted from the streak shown in C. Note the clearly defined octahedral cleavage. Photographed in water by incident light, with a 4 mm. water immersion objective, × 300 diams.

E. Fragments of refractory chromite as seen in a streak similar to that illustrated in C, after a light surface polish. This print has been rotated through 90° relative to the streak. Octahedral cleavage is again present. Incident light, × 300 diams.

Addendum. Booker *et al.* (1960) carried out a comprehensive examination of aluminous inclusions in iron, employing a replica extraction method previously used by two of the authors. Examination was by incident light, transmitted polarized light, electron microscopy, and X-ray diffraction.

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