

Acceptance tests for steelworks refractories

J. H. CHESTERS

REFRACTORIES represent a substantial part of the costs of converting iron into steel, varying greatly from a few shillings to more than £1 sterling/ton of steel. Their quality is, therefore, of considerable importance: even a 10% improvement represents a substantial saving. Equally important, any serious deterioration, involving, say, the premature collapse of a furnace roof, or the breakout of steel through a hearth, not only means extra cost but frequently a plant stoppage. The quantities of materials involved are illustrated by figures given for the third Five Year Plan¹ for India. Table I shows the likely demand for refractories and the capacity considered necessary to supply the whole of industry.

It is suggested that the difference could best be met by the installation near steelmaking plants of additional brickmaking units of 20 000 to 30 000 tons/a capacity. These would appear typical of the present average size, there being some 42 refractories plants in production in 1960. What is surprising, almost alarming, are the figures for refractories consumption on which these estimates are based. These are given in Table II.

If these consumptions, which exclude cements and magnesite used for ramming, could be reduced to figures obtained with steelmaking in other parts of the world, the proposed expansion would be largely unnecessary. Just how much of the high consumption is the result of low brick quality, and how much of plant usage, would require to be investigated, but periodic testing is clearly desirable, if only to exercise some measure of control over such a major expenditure.

One hundred per cent testing, or even inspection, is out of the question in view of the large number of units handled. Rough calculation suggests that in a steelworks making 1 m. tons/a, three men testing 1% of the bricks during a 40 h week would each require to produce complete test data on some 20 bricks/h! This is clearly absurd. Even with a substantially larger staff and some measure of automation in testing, sampling and testing rates of more than, say, 1 in 10 000 units are likely to prove impracticable. In view of this dilemma, recourse must be had to periodic sampling, which should ideally be carried out on a regular, and preferably, statistical, basis. In fact the tendency will always be to test most frequently the new or unreliable product and only occasionally the regular supply. This means occasionally 'closing the stable door after the horse has gone', but even this is worthwhile if it results in the stable door not being left open so often.

SPECIFICATIONS

We would not claim to know whether these are, on balance, a good thing but we would congratulate India on having pushed on so vigorously in preparing her own standards, both for test methods and products. There is no doubt that those prepared for moderate heat duty and high heat duty fireclay bricks, and bottom pouring refractories, including sleeves and ladle bricks, will ultimately be of considerable value, if only as standards by which achievement can be judged. Simple logic would

SYNOPSIS

Refractories represent a substantial proportion of conversion costs and may, if unsatisfactory, lead to lost production. These facts, together with the relatively high consumption of refractories per ton of steel in India, emphasize the need for acceptance testing. In the absence of international standards, specifications should be limited to known essentials, and regular supplies only tested periodically once satisfactory background data are available. New supplies should always be tested, and in addition material whose appearance or service behaviour suggests poor quality. Equally important is the establishment of satisfactory user-supplier relations, with the associated exchange of data. The equipment required for acceptance testing is not particularly expensive, and the work in a 1 m. tons/a plant can, it is suggested, be handled by one ceramics engineer and two assistants.

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suggest that specifications be prepared for all types of brick and that those not meeting them be rejected. Certain firms, notably in the USA, have gone part way towards this target, asking a range of suppliers to quote to a specification and provide at the same time information on other properties. When a contract is placed, the user apparently reserves the right to reject not only material that is outside specification but bricks that do not come up to the general standard described by the suppliers in their quotation. The alternative approach, and one which we would favour, can be summarized as follows:

- (i) each plant maintains a small staff, equivalent to about three per million tons of steel made, who watch over refractory deliveries and their performance. These are additional to a central staff carrying out research and development
- (ii) close contact is maintained with the supplier, so that the benefit of his larger quantity of routine data be obtained
- (iii) (a) suspect materials are immediately tested, e.g. bricks that are obviously weak or laminated, or behave poorly in service
(b) with new products a whole range of properties is determined in order to get a general impression of their quality and also background data for future reference
(c) periodic checks are made on the principal lines purchased, e.g. silica bricks and dolomite.
- (iv) any specifications used are limited to simple statements such as 'super-duty silica bricks shall contain less than 0.5% alumina', the maintenance of other properties being left to the pride of the supplier.

In adopting this rather loose approach, we are considerably influenced by a feeling that a specification can all too easily become a boomerang. Thus definition of super-duty silica brick in the US, as 'containing less than 0.5% of alumina+alkalis+titanium', excludes one of the best super-duty bricks developed in the UK because, though low in alumina, it contained about 1½% of apparently harmless titanium. Difficulties can also arise because material though well within specification is nevertheless unsatisfactory owing to an unfortunate characteristic not covered by the specification. Thus for a long while we

The author is deputy director of research, The United Steel Companies Ltd, Swinden Laboratories, Moorgate, Yorkshire, England.

TABLE I Inidan refractories demand and capacity

Type	Likely demand (tons)	Required installed capacity (tons)	Installed capacity 1960 (tons)
Fire bricks	8 00 000	1 012 000	520 000
Silica	3 50 000	434 000	70 000
Basic	1 80 000	227 000	44 000
High alumina	54 000	68 000	10 000
Miscellaneous	7 000	8 800	6 400
Dead burned magnesite	90 000	112 500	64 320
Fire cement and mortar	1 39 000	174 000	85 800
	16 20 000	2 037 000	8 00 520

thought the vital feature of high-frequency linings to be low shrinkage at steelmaking temperatures, whereas steel-tight linings were ultimately achieved by insisting on a positive expansion around 1 150°C.²

PRODUCTS CONSIDERED

The following discussion is limited to some of the more important refractories used in steelworks, e.g. silica and basic roof bricks, ordinary silica bricks, fettling dolomite, and casting-pit refractories, including ladle bricks.

Silica roof bricks

Two tests are generally sufficient for control purposes, namely chemical analysis, and in particular, alumina content and porosity. The latter yields automatically the bulk density and the apparent solid density which, with silica, is virtually identical with the specific gravity. There is still some difference of opinion as to whether a really hard-fired brick, as generally used in the UK (2.32 to 2.34 sg) or a softer-fired brick (up to 2.40 sg) as frequently used in the rest of Europe, is better, but all users would join in deploring wide variations in specific gravity or excessively high figures (2.50 or over) such as lead to uneven or excessive growth.

This was well illustrated by a weekend repair in one of our plants some years ago. The manager complained on Monday morning that his instructions had not been followed, in that a backwall had not been replaced. He was told that it had in fact been completely rebuilt but that the permanent growth on heating-up was so great that it had almost all sheared away. Subsequent tests showed the bricks to have a specific gravity of about 2.50 and to consist largely of raw quartz. The bricks had not been bought to specification but were nevertheless rejected as of quite inadequate quality. Such rejections are usually accepted gracefully, the supplier knowing that failure to replace faulty material is unlikely to be good for future business.

Periodic tests, both of alumina and specific gravity, can, when collected together, provide a quality control picture, revealing for example the effect of such changes as washing of the raw rock or kiln practice. One histogram showing a number of samples of different specific gravity had a peculiar double-hump structure, suggesting two statistical 'populations'. Enquiries showed that the manufacturer was using both round and square kilns and that the bricks located in the corners of the latter were unusually soft-fired. This led to future deliveries of roof bricks all being fired in round kilns.

Basic roof bricks

In spite of the vast amount of research done on basic bricks, few technologists would dare to issue a rigid

TABLE II Consumption of refractories/ton of steel in silica and in all-basic roof practice

Type of refractories	For silica roof (lb)	For all-basic roof (lb)
Fire bricks	119.06	80.00
Silica	51.76	25.00
Basic	27.05	45.00
High alumina	7.56	10.00
Other types (insulating, carbon, silicon carbide, etc.)	1.57	5.00
	207.00	165.00

purchase specification for all-basic furnace roofs. Having tested virtually every basic roof installed in our plants for many years, we reached the conclusion that the three most important features of fired chrome-magnesite bricks were their silica content, their permanent change on reheating (less than 1% after 2 h at 1 700°C) and their hot strength, as measured in torsion³ at 1 300°C. As the general level of brick quality has risen, the correlation between even these properties and roof life has largely disappeared, until today silica content alone appears to correlate significantly. It would be easy to write a specification for a chrome-magnesite brick, giving maximum porosity, minimum cold crushing strength, fail point in refractoriness-under-load test and, say, thermal shock resistance, but its application, though eliminating the really inferior product, would still be of little value in selecting the best. Thus many workers believe that the amount of bursting that occurs when a chrome-magnesite brick is heated up in contact with iron oxide is a vital factor, but although we still believe iron oxide to be a major cause of damage, we find no correlation between bursting index and roof life with high grade bricks. Similarly India has produced some excellent chrome-magnesite bricks by stabilizing the high iron-chrome ores with magnesia. Our specification of low silica content would certainly exclude all of these, though I gather that they are definitely an economic proposition.⁴

Other basic bricks

The standards demanded of chrome-magnesite bricks used in positions other than roofs are less stringent and it might, therefore, be expected that in the absence of specifications producers would tend to put inferior material into standard squares, to compensate for the use of specially selected material for roofs. It is encouraging to note, therefore, that a recent survey made by us on the chrome-magnesite bricks available in the UK, not only showed that the quality of ordinary squares was very good but that it was actually higher than that of roof bricks of a few years ago. Under such conditions close controlled testing of ordinary chrome-magnesite bricks would be a waste of effort.

Other silica bricks

Here again standards are not as severe as for roof bricks, though attention to specific gravity, bulk density, and alumina content is still desirable. Here also the dangers of buying to specification are illustrated by the recent use in India of a specification for silica bricks that makes no mention of alumina content but only of silica as a minimum of 96%. Still more surprising, second-class bricks are quoted as having a higher silica content than first-class bricks, the main difference between the two apparently being the degree of firing, which in turn apparently affects the refractoriness-under-load.

Fettling dolomite

Assuming the dolomite to be reasonably pure, the points requiring attention are grading, loss on ignition on the fine fraction, and bulk density of the individual dolomite grains, the latter providing a good index of firing treatment.⁵ The ultimate grading of the material, whether used for bottoms or fettling, is of course vital. Badly hydrated material is such a nuisance that certain firms reject all fines on arrival, while bulk density is generally worth paying for. Boddy,⁶ in a recent paper on the VLN process, shows a remarkable correlation between the bulk density of dolomite and the bottom life achieved.

Casting-pit refractories

Failure here can easily result in loss of considerable quantities of steel or, equally serious, in non-metallic inclusions. It is not, therefore, surprising that casting-pit refractories were among the first included in Indian specifications. Much can be learned by visual inspection, e.g. accuracy of dimensions, freedom from warpage, or ovality, and the presence or absence of black cores. Where check tests are done these will clearly include porosity, thermal shock resistance, permanent linear change on reheating, cone melting point and, occasionally, chemical analysis. Considerable value is attached to a periodic survey of the materials in use. This not only highlights potential weaknesses but provides a basis for future comparison. Thus if trouble is experienced due to spalling of mould bricks, and their thermal shock resistance, as tested in the laboratory, has been found to fall, the only remaining question is how this has happened. One particularly serious case was found to have arisen by mould top bricks being fired in the same kiln with sillimanite, which required a particularly high firing temperature. Thermal shock tests proved the deterioration, and X-rays were used to estimate the unusually high firing temperature employed. Here again there was no specification, but the supplier agreed to remove the offending material.

The difficulties of specifications might be further illustrated by the Indian Standard, IS. 525, 1960, which refers to ladle bricks. With the clays available in India this may be a rational standard, but it is perhaps worth noting that it would result in the rejection of some of the best bricks in the world, e.g. the bloating type extensively employed in the USA, whose cone melting point is only 20 (1 530°C) compared with cone 30 in the Indian specification. Incidentally it is not clear to us why different specifications should be required for ladles below and above 100 tons. A 500-ton ladle recently observed in the USA was said to give no more trouble with ordinary bricks than one of much smaller size.

EQUIPMENT AND TEST METHODS

The first clearly depends on the second, and unfortunately each of the larger nations seems to have its own test procedures, see for example the ASTM, BSI, DIN, and USSR, not forgetting the Indian standards (IS 485, to 1953). In carrying out tests the user will, of course, use his own particular equipment and there are bound to be arguments with suppliers using their own national standards. In some cases these will have to be subject to referee testing. The difficulty is well illustrated by a paper by Banerjee and Nandi,⁷ who compare the results obtained in testing Indian refractories according to different specifications. They were at the time particularly concerned with the refractories to be used at Bhilai. Since this plant was being built by the Russians, they carried out tests on high and moderate heat duty

firebricks and on silica bricks, using Indian and Russian standard methods. As a result they suggested certain minor changes in the Indian specification. Had the work been done at Rourkela or Durgapur the comparison would presumably have been made with DIN and BSI tests, in which case other changes would doubtless have been recommended. The only real solution to this problem is of course international methods of test. These are already under development, but progress is extremely slow, mainly because of the endless minor compromises needed to achieve agreement. A major problem is of course that changing one's standards means that new test data are no longer comparable with those already on file. That this need not, however, be a major problem is shown from our own experience in changing from company to national (British) standards when the latter first became officially available.

Being in the process of installing more systematic testing of refractories in each of our main steelmaking branches, we have recently given some thought to the minimum equipment required. Our conclusions are summarized in Appendix I, which it is hoped may be of value to other workers wishing to set up a similar degree of control. The approximate cost of this equipment in the UK is £3 000 apart from the refractoriness-under-load equipment, which may cost as much as £2 000 extra.

Although the final responsibility for producing the desired refractories lies with the manufacturer, he can be greatly assisted if the results of routine and special testing are communicated to him and discussions held regarding possible lines of improvement. It is usually the refractories technologist in the steel plant who knows best why the material has failed and, therefore, what changes in properties are likely to result in an improvement. A vast amount of information is now available on the factors controlling brick properties and the effect of these properties on service performance. We are, however, abysmally ignorant of many vital facts and must, therefore, adopt a humble attitude throughout. Rigid specifications imply firm knowledge, and it is mainly for this reason that we suggest they be limited to essentials, the general question of quality being left to friendly discussion between maker and user.

CONCLUSIONS AND RECOMMENDATIONS

1. Acceptance testing by steelworks is favoured but specifications should be limited to known essentials.
2. Regular supplies need only be tested periodically once full background data are available.
3. New supplies should be tested to obtain a picture both of their general quality and variability.
4. Special tests should be made on material that appearance or service suggests is inferior.
5. User-supplier relations should be built up by exchange of test data, including service performance.
6. The refractories test equipment needed for a plant making a million tons of steel need not be expensive, and the work involved can be done by one ceramics engineer and two assistants.

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APPENDIX I

EQUIPMENT REQUIREMENT

Equipment	Tests	Recommendations
Cutting machine	All tests on solid specimens	Cutting machine, e.g., diamond rimmed wheel. A drilling machine with diamond tipped core drills is a useful addition and may ultimately offer economies in specimen preparation
AFA rammer	Packing density tests and production of test specimens of monolithic refractories	The AFA rammer and necessary moulds for producing 2in dia. compacts
Crushing and grinding	Chemical analysis	Small laboratory jaw crusher, iron pestle and mortar, a hardened steel percussion mortar and an agate pestle and mortar would be ideal ; preparation is possible but laborious without the jaw crusher
Vacuum dessicator and pump	porosites and densities	12in dia. vacuum dessicator and vacuum pump
Balance	Porosity and densities, moisture content. Gradings, bulk densities, weighing of volumenometer specimens	Capacity 250g. Accurate to 0.01g ; Capacity 1 000g. Accurate to 0.1g

Caliper gauge	Permanent change on reheating. Drying and firing shrinkage	Vernier caliper with 12in capacity between jaws reading to 0.001in
Mercury volumenometer	Permanent change on reheating	e.g. : apparatus described in <i>Trans. Brit. Ceram. Soc.</i> 49.305
Mercury volumenometer	Bulk density of dolomite	Apparatus described in <i>ISI Spec. Rep.</i> 33, p. 21
Sieves	Gradings and sample preparation	e.g. : 8in BS sieves of 1/2in, 1/4in, 7, 10, 25, 72, 150 mesh
Pyrometric cones	Refractoriness	Standard Orton cones are recommended
Warpage gauge	Warpage	Apparatus described in connection with ASTM standard test C. 67-57
Moulds	Preparation of specimens of castable and mouldable refractories	Demountable mould to yield specimen 15in x 6in x 3in
Compression machine	Transverse strength	A universal or compression machine with at least 10 tons capacity is suitable
Furnaces	Permanent change on reheating and other high temperature tests	High-temperature furnace capable of at least 1700°C and heating rates up to 5-6 deg C/min. from 1500°C to 1700°C. Gas firing is recommended
	Thermal shock resistance and medium temperature reheat tests	Medium temperature furnace of high heat capacity. Gas firing is recommended
	Bend tests on basic roof bricks	Resistor bar furnace with uniform temperature distribution within chamber 24in x 10in x 9in
Temperature recorders	All high temperature tests	Electronic potentiometric recorders for appropriate ranges