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A new method for the rapid float–sink analysis of coal fines

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SYNOPSIS

Float-sink analysis has wide application in the coal laboratory. This paper evaluates the established methods used for the float-sink analysis of fine coal, and then describes a new method and apparatus that can be used for material down to a few micrometres in size. The method is rapid and accurate, and requires only a small amount of sample. The results presented show close agreement with the float-sink analyses obtained by the more classical technique using separating columns. The new centrifugal method reduces the time required for the analysis by a factor of ten.

SAMEVATTING

Gravitasie skeiding analises het 'n wye toepassing in die steenkool laboratorium. Hierdie referaat evalueer bestaande gravitasie skeiding analise tegnieke vir fyn steenkool en beskryf dan 'n nuwe metode en aparaat wat vir partikel groottes van slegs 'n paar mikrometers gebruik kan word. Die metode is vinnig en akkuraat en benodig slegs 'n klein hoeveelheid monster. Resultate, wat goed ooreenstem met dié verkry in die meer klassieke skeidingskolomtegniek, word ingesluit. Die nuwe sentrifigale metode het gely tot die reduksie in analise tyd met 'n faktor van tien.

Introduction

Float–sink analysis is a widely used technique in the coal laboratory. It finds application, firstly, in the study of coal washability or liberation from ash and, secondly, in the determination of coal-washery performance through the use of partition curves and the data derived from them. However, while float–sink analysis of plus 0,5 mm material presents little difficulty, the established methods are tedious and tricky for fine coal. This is particularly true of coal smaller than 53 μm in size.

Existing Methods

The most common method for the float–sink analysis of fines involves the use of separating funnels in which particles are separated under gravity in a suitable heavy liquid. For samples containing very fine particles or a high proportion of near-gravity material, this technique becomes extremely time-consuming since a very long separating time is required. Also, the separation of coal particles finer than 53 μm by this method has been found to be impracticable[1].

Alternative methods aimed at speeding up the process rely on the use of a centrifuge. In the method recommended by the International Organization for Standardization (ISO)[2], a sample of coal (20 to 60 g) is placed in a centrifuge tube with liquid of the lowest density. After spinning, the floats are collected from the surface with a scoop, liquid of the next highest density is added to the sinks, and the process is repeated until the highest density is reached. A disadvantage of this method is the initial high loading of the tubes, which can result in a significant amount of entrained, and thus misplaced, material; moreover, it is difficult in practice to scoop all the floats from the surface accurately without leaving some material behind or causing some mixing with the sinks. The sequential treatment of a single sample can also lead to significant cumulative error as the sample being treated becomes smaller.

Another centrifugal method is that of Hall[3], in which a fine gauze tray is fitted inside the centrifuge tube, being suspended between the floats and the sinks by wires hung over the edge of the tube. This gauze is used to pull out the solid plug of floats after centrifuging. Problems with this method are that the gauze, during the centrifuging, tends to trap some sink material, which will report to the floats; and that, when the gauze is removed some of the float material is invariably left behind and is difficult to recover.

New Apparatus and Method

A new apparatus has been devised for speeding up the float–sink analysis of coal fines, and making it less prone to error. This apparatus, which is also designed to fit into a centrifuge tube, has been provisionally manufactured out of Perspex, although, as this is soluble in most organic liquids, a material such as polytetrafluoroethylene (PTFE) or stainless steel would be more suitable.

The heavy liquid used to date is zinc chloride. Its advantages are that it is very cheap, and does not give off toxic vapours like the more commonly used organic liquids such as bromoform or tetrabromoethane (TBE). A limitation is that the highest relative density that can be obtained is 1,8.

The device, shown in Fig. 1, fits inside a standard 100 ml PTFE centrifuge tube. It consists of a tapered

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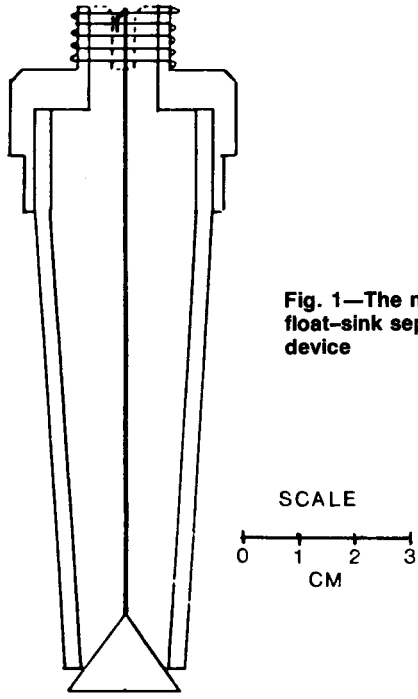


Fig. 1—The new float–sink separating device

tube, with the bottom sealed by a conical plug. This plug is connected via a rod to a spring at the top of the device. When sufficient downward force acts on the plug (as occurs during centrifuging), the spring is compressed, opening the bottom of the tube.

The use of the new apparatus is illustrated in Fig. 2. Unlike the ISO method, in which a single sample is treated sequentially, the new method uses separate samples at each relative density, obtaining cumulative data directly.

The procedure can be broken down into four stages.

A Approximately 2 g of dry coal is accurately weighed into a 30 ml sample bottle. The sample bottle has a conical bottom leading into a 4 mm glass tube, which is connected to a short piece of rubber tube. The rubber tube is clamped shut. A few millilitres of zinc chloride solution are added to the bottle, along with a few microlitres of a chemical dispersant known as Tween 20 (polyoxyethylene sorbitan monolaurate). After the bottle has been sealed, it is shaken until the coal is thoroughly wetted. The bottle is then placed in an ultrasonic bath for 5 minutes to aid in further dispersion. The lid of the sample bottle is removed, and any adhering coal is washed into the bottle with a few millilitres of zinc chloride solution. The rubber tube of the sample bottle is then inserted into the top of the separating device, and the clamp is removed. A syringe is used to wash any remaining coal particles from the sample bottle into the device. The separating device is then filled to the required level with zinc chloride solution, as is the centrifuge tube.

B The device is inserted into a centrifuge tube, although the contents of the two remain isolated from each other. The tube is then spun in a centrifuge.

C During spinning, centrifugal force on the conical plug pulls it down, compressing the spring. This links the fluid in the separating device with that in the centrifuge tube, and enables sink material to enter the tube.

D When the centrifuge stops spinning, the spring causes the conical plug to seal the device again, isolating the float material from the sink material. Thus, the two fractions are separated by mere removal of the device from the centrifuge tube. The separation is achieved without any of the problems associated with the methods previously discussed.

It is important that a centrifuge with a swing-arm rotor should be used, so that the line of force acts down the centre of the tube. With the tube spinning at 4000 r/min, about 15 minutes are required for samples of material larger than 25 μm; samples of smaller material usually require between 1 and 2 hours of spinning time.

If material smaller than 53 μm is being analysed, the conical plug can be locked during the initial centrifuging, resulting in a provisional separation in the device. If this is not done, the fine coal slurry in the device would behave like a liquid with a higher relative density than the clear zinc chloride solution in the centrifuge tube. Thus, when the conical plug opened, mixing would result, causing some float material to report to the sinks. After a provisional separation has been obtained, the spring can be released, enabling the conical plug to open, and the tube can be re-centrifuged to give the final separation.

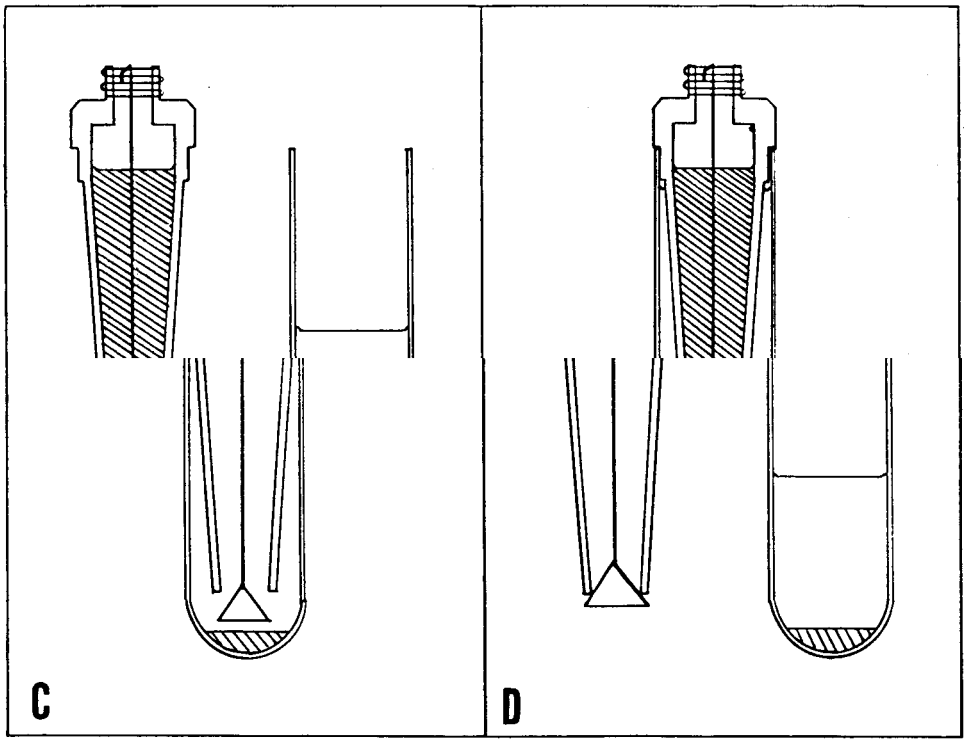
Reproducibility of Results

In order to establish the precision of the method, a number of replicate experiments were conducted on different size fractions of a sample consisting of a synthetic mixture of washed and waste coal from the Greenside Colliery. The float–sink analyses were carried out at relative densities of 1,4, 1,5, and 1,6. As can be seen from Table I, the method is extremely precise for the size ranges examined, the maximum standard deviation for four replicates being only 0,6 per cent. Also, a mass balance of the float and the sink material shows acceptably small and relatively constant losses in mass.

TABLE I
REPRODUCIBILITY OF THE NEW METHOD

Size fraction μm	Relative density	Number of replicates	Mean float %	Standard deviation	Average lost material %
−300 + 106	1,6	4	56,3	0,2	0,9
−106 + 75	1,6	2	59,1	0,3	2,8
− 75 + 38	1,6	4	59,9	0,3	2,5
− 38 + 20	1,5	4	49,8	0,6	2,1
−300 + 106	1,4	4	34,5	0,4	2,4
−106 + 75	1,4	2	34,8	0,2	2,4
− 75 + 38	1,4	4	31,1	0,5	2,5

To investigate the reproducibility of the method, samples of Greenside No. 2 seam coal were obtained from Professor R.P. King of the Department of Metallurgy at the University of the Witwatersrand. The samples consisted of cyclone underflow and overflow that had been taken during the operation of a pilot rig in which the dense-medium separation of fines was being investigated[4]. Partition-curve data were obtained by the new method for the cycloning of material between 90 and



75 μm. The results were then compared with those of Professor King's research group, which had been obtained by use of the double-column gravitational separating technique illustrated in Fig. 3. This method is very accurate but extremely time-consuming. The top column acts as a 'rougher' and the lower column as a 'cleaner'. Sequential treatment of a single sample was used, with Certigrav as the heavy liquid.

Partition curves comparing the two sets of results are shown in Figs. 4 to 6. (The horizontal axes—labelled 'Mean Specific Gravity'—would be more accurately described by 'Mean Relative Density'.)

The following partition function used by King[4] was fitted to the results:

$$R(x) = b_3 + (b_4 - b_3)\frac{\exp(b_2 x) - 1}{\exp(b_2 x) + \exp(b_2) - 2},$$

where x = normalized relative density, S/S_c
S = relative density of particle
S_c = cut-point relative density (S at a partition factor of 0,5)
R = partition factor
b_2, b_3, and b_4 are function parameters.

TABLE II
CURVE-FITTING DATA FOR PARTITION CURVES 1, 2, AND 3

Item	New method	Funnel method	Combined data
Partition curve 1 (Fig. 4)			
S_c	1,563	1,57	1,57
$\Sigma(\text{error})^2$	42,30	21,32	73,56
$\frac{\Sigma(\text{error})^2}{\text{No. of points}}$	6,04	2,13	4,33
Partition curve 2 (Fig. 5)			
S_c	1,546	1,553	1,550
$\Sigma(\text{error})^2$	34,46	198,48	247,55
$\frac{\Sigma(\text{error})^2}{\text{No. of points}}$	4,96	19,85	14,56
Partition curve 3 (Fig. 6)			
S_c	1,519	1,523	1,523
$\Sigma(\text{error})^2$	121,09	187,17	386,43
$\frac{\Sigma(\text{error})^2}{\text{No. of points}}$	20,18	20,78	25,76

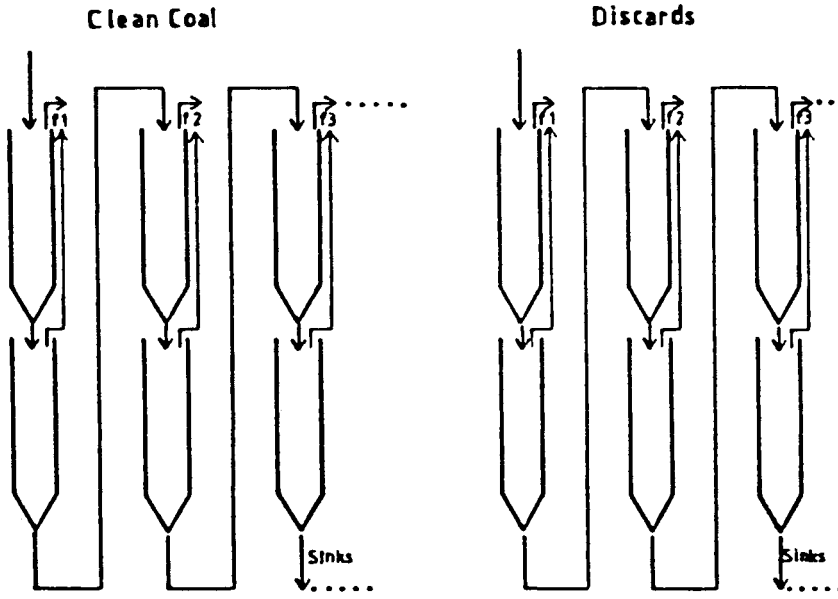


Fig. 3—Double-column method of float–sink analysis (from King and Juckes[4])

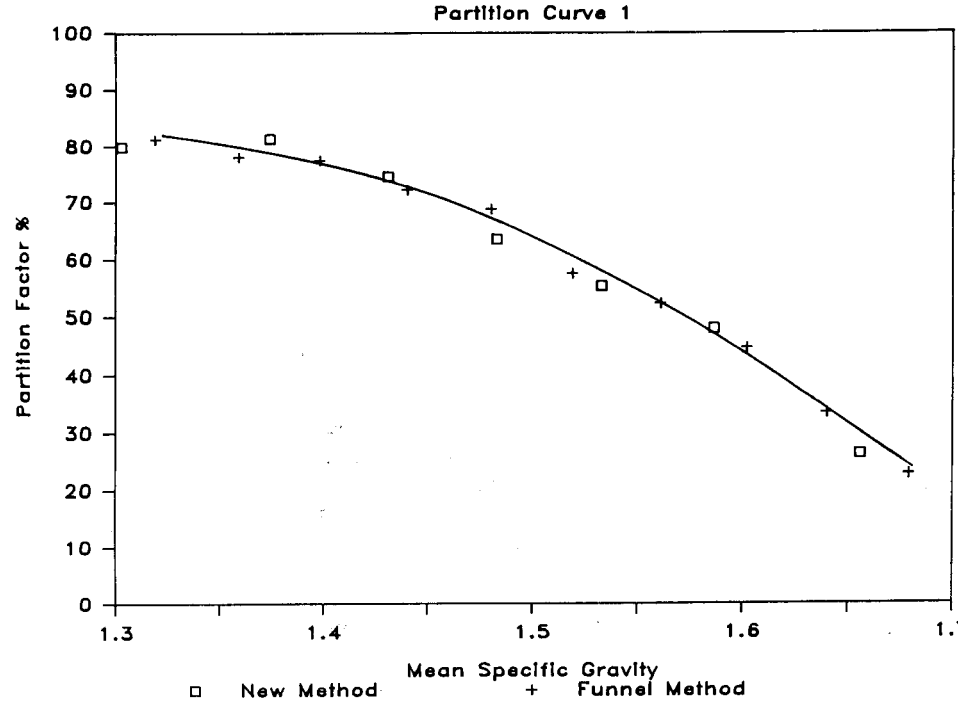


Fig. 4—Comparison of the results obtained from the new method with those from the double-column method, sample 1

Least-squares best estimates of the parameters S_c, b_2, b_3, and b_4 were obtained by non-linear regression analysis of the experimental data.

In each case, curves were fitted to the combined set of data, and to each set of data obtained by the two different methods. Table II presents the least-squares fit for each curve and the cut-point. As a different number of points was used to obtain each curve, the sum of error squared divided by the number of points is included for purposes of comparison.

A very good agreement between the two sets of data can be seen in Figs. 4 and 5. The poor fit apparent in Fig. 6 can be explained by the fact that there was insufficient material to obtain more points in the higher density range of the graph (see Fig. 6, graph A). Thus, the first two points have an exaggerated effect on the shape of the curve. Both sets of data exhibit a high degree of scatter, indicating possible inconsistencies in the coal sample, rather than problems in either of the float–sink methods. The high degree of scatter requires a large

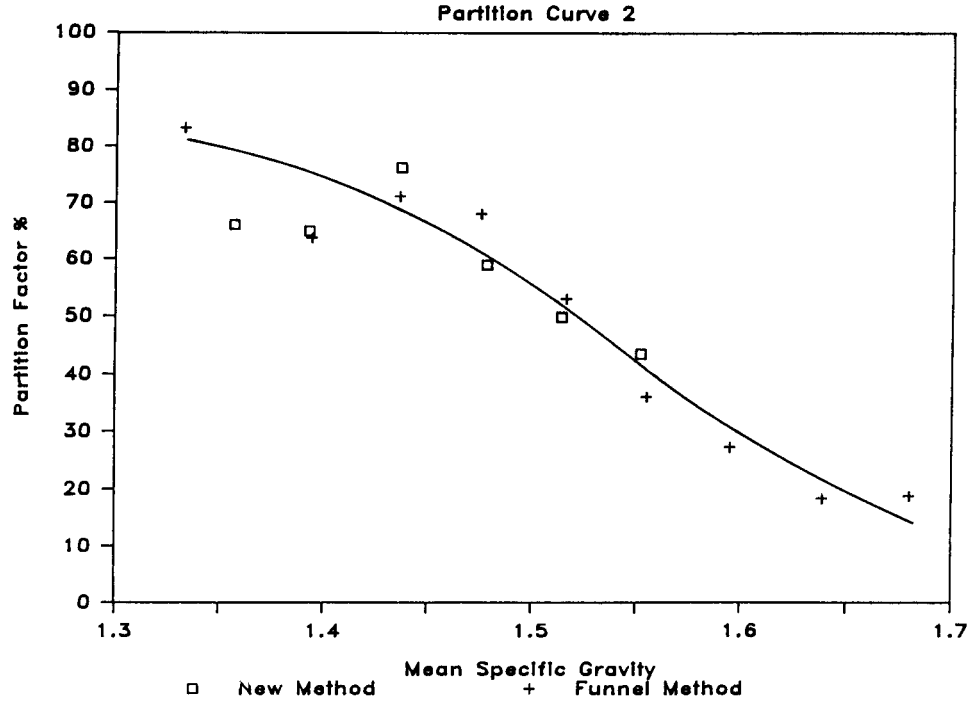


Fig. 5—Comparison of the results obtained from the new method with those from the double-column method, sample 2

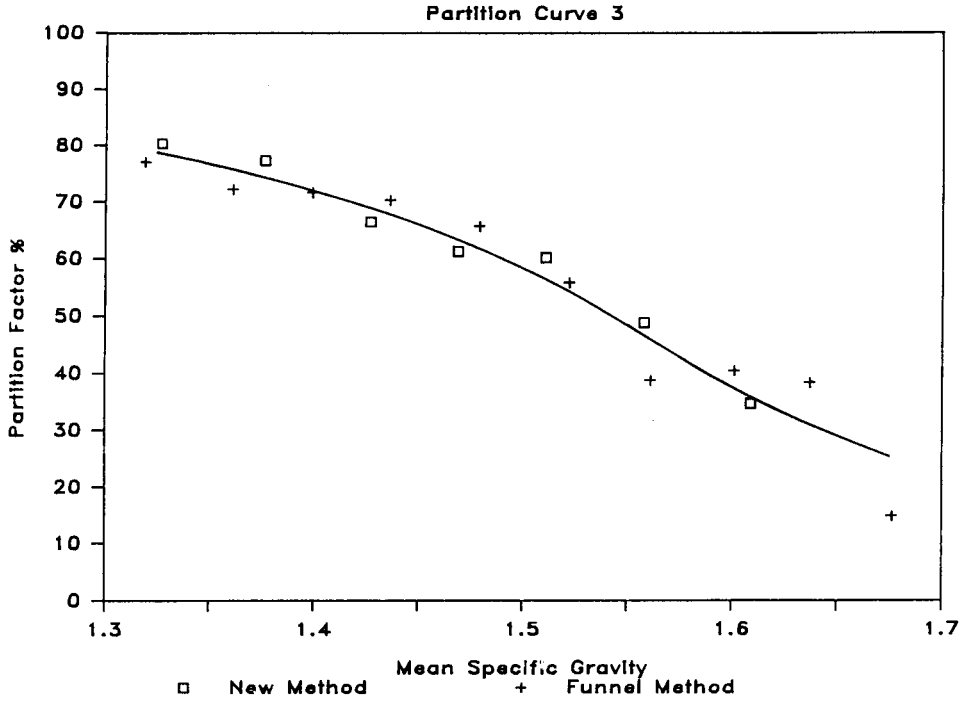


Fig. 6—Comparison of the results obtained from the new method with those from the double-column method, sample 3

number of points if the curve fitting is to be satisfactory. When the graph based on the combined data is compared with that based on the separating-funnel data, a much better agreement is apparent.

Conclusions

Float–sink analyses can be carried out very easily with the new apparatus, with none of the problems associated with other methods. The method has been shown to be reproducible, and its results correspond satisfactorily to the results obtained by the more traditional separating-funnel method.

On account of the difficulties associated with other methods, many collieries and coal laboratories do not carry out float–sink determinations on the minus 0,5 mm fines, despite the increasing proportion of this size fraction in run-of-mine material. The simplicity of the new separating device and method makes it possible for such analyses to be made quickly and accurately on a routine basis.

Although the apparatus was designed specifically for use in the beneficiation of fine coal, its application is not limited to coal, and it should prove a useful tool in all mineral-processing laboratories.

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Flat rolling

The 4th International Steel Rolling Conference is to be held in Deauville (France) from 1st to 3rd June, 1987.

The Conference will provide an opportunity for specialists to make an appraisal of the status and trend of development of the process and operating technology of rolling on plate, and hot- and cold-strip mills. The scope of this event will extend to all stages of the process, starting from the hot slab ready for rolling down to the temper-mill for cold-reduced or hot-strip mill material. The metallurgy of continuous annealing will not be explored.

In the case of the plate/mill, heat treatment such as rapid cooling out of the rolling heat is clearly part of the process, although the handling of such topics should concentrate on the thermal and technological aspects of this particular process and on its consequences on the upstream and downstream operations, and not on the metallurgy; hot and cold levelling will, of course, be considered.

New mill strand and roll technology should be reviewed, and their effectiveness in controlling strip shape and easing the hot strip mill scheduling constraints appraised on the basis of theoretical and experimental work.

The influence on plant efficiency and product quality of the coupling of several stages of the process and of the processing of 'endless strip' in the cold-rolling plant should also be further analysed.

Mill computer control based on accurate mathematical models remains an essential area for research and development, while closed-loop control linked to recently introduced sensors and actuators, which has found new applications in the field of flat rolling, deserves the attention of the specialists.

Although it is not intended to include in the programme reheating furnaces, coating lines, nor product metallurgical developments *per se*, papers dealing with such topics as the following fall within the scope of the Conference:

- Influence of the product temperature level and homogeneity on mill control, mill performance, and product quality
- Optimum use of the technical features of the rolling plant in relationship to metallurgical and surface-finish requirements.

The design of production scheduling systems may also be considered in so far as the emphasis is placed on such desired impacts on plant operation and mill performance as

- Schedule free rolling on the hot-strip mill
- Hot charging and direct rolling
- Improved crown and flatness control of the hot-rolled strip.

Lastly, maintenance, which has such a direct influence on plant efficiency, should not be overlooked.

Further information can be obtained from

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