May 6, 1969 K. G. CARR-BRION ET AL 3,443,092

CONTINUOUS X-RAY FLUORESCENT ANALYSIS OF THE SOLID COMPONENT OF A SLURRY

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Fig. 1.

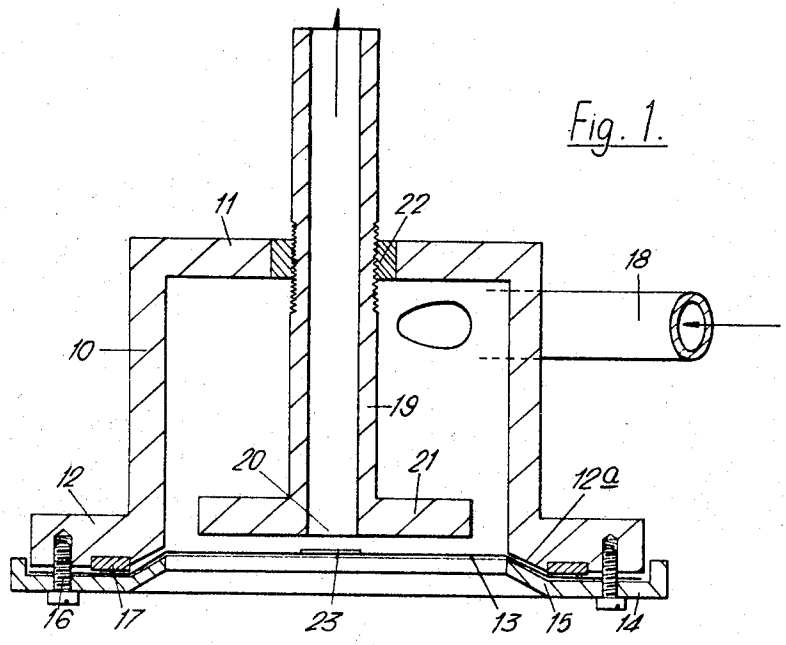
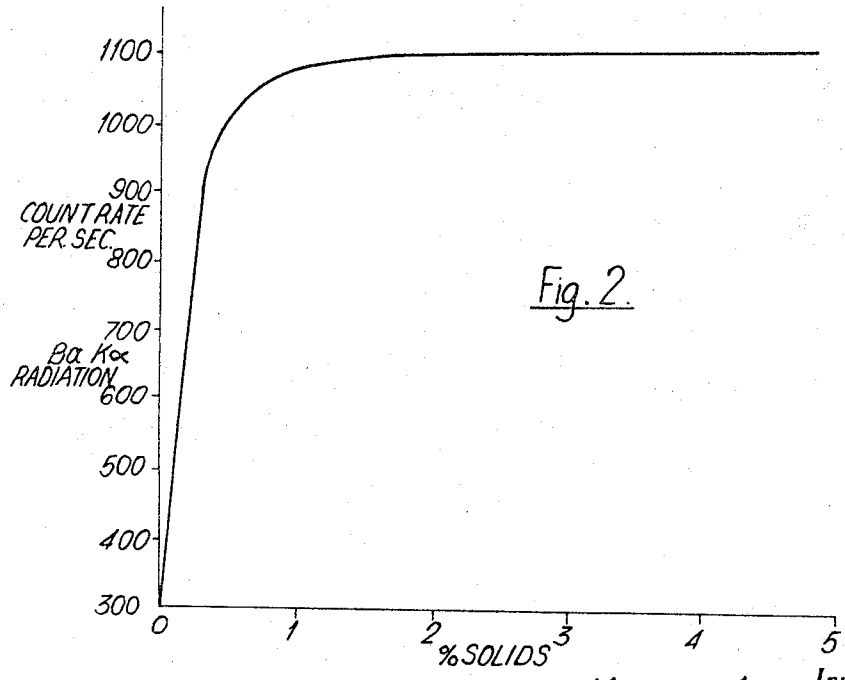


Fig. 2.



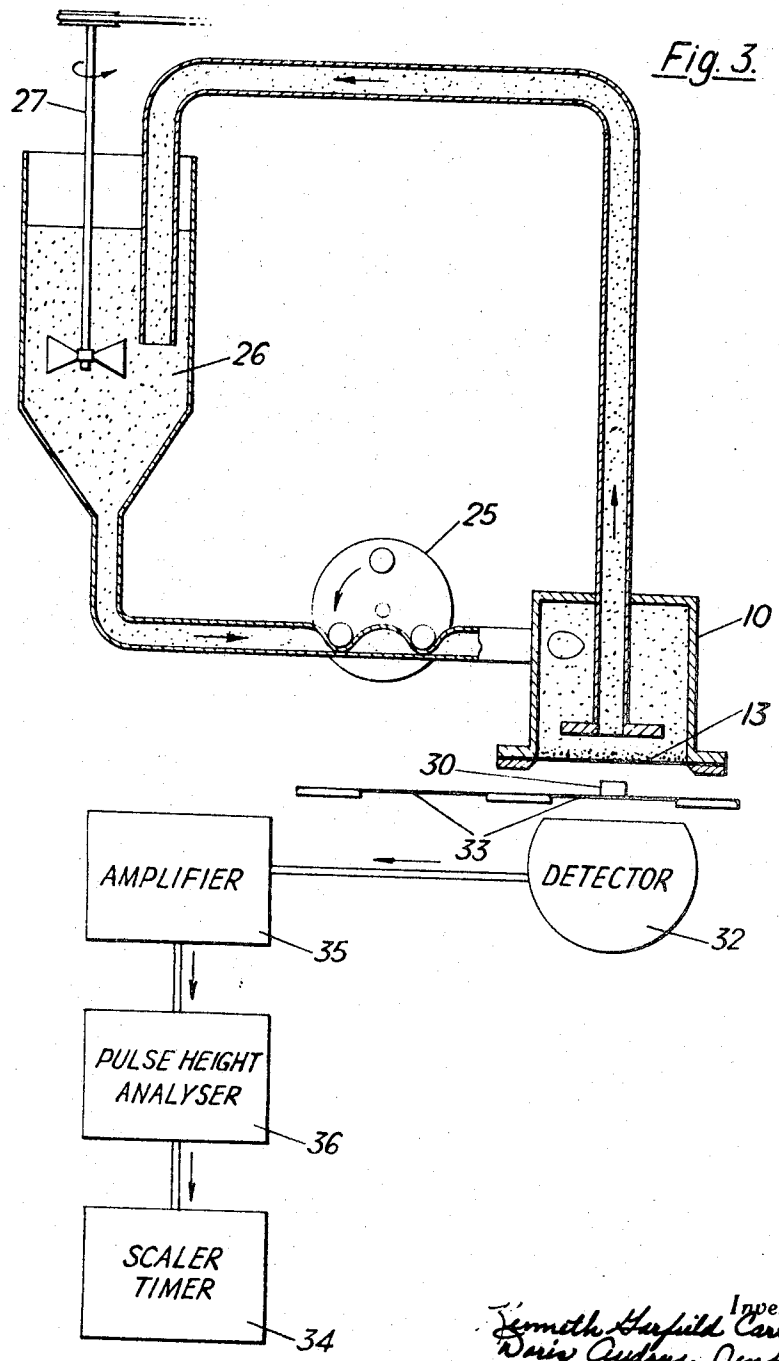
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Fig. 3.



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CONTINUOUS X-RAY FLUORESCENT ANALYSIS OF THE SOLID COMPONENT OF A SLURRY

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U.S. Cl. 250—51.5
15 Claims

ABSTRACT OF THE DISCLOSURE

Method and apparatus for testing continuously the concentration of a given element in the solid component of a slurry in which the solid component is at least partially separated centrifugally from the slurry, the solid component so separated being moved in a continuous layer across and in contact with a window of said apparatus, and the solid component in said layer being analyzed by means of radiation transmitted through said window.

This invention comprises improvements in or relating to the continuous analysis of the solid component of a slurry. The handling of solids in many manufacturing processes is assisted by suspending the solids in a liquid slurry. Where it is desired to perform a continuous analysis of the solids in the slurry as a way of, for example, monitoring some stage of a manufacturing process to control the same, some arrangement for presenting the solid component of the slurry independently of the liquid component is desirable. This is particularly true, for example, where the analysis of the solid component is performed by an X-ray fluorescence technique, since the result of the analysis must otherwise be corrected for the concentration of the solids in the slurry, a correction which requires a precise knowledge of the density of the slurry and the solid component, which is in many cases difficult to obtain.

The present invention provides, in one aspect, a method of testing continuously the concentration of a given element in the solid component of a slurry in which the solid component is at least partially separated centrifugally from the slurry, the solid component so separated is moved in a continuous layer across and in contact with a window, and the solid component in said layer is analysed by means of radiation transmitted through said window.

Preferably said analysis is effected by inducing X-ray fluorescence of the solid component in said layer and analysing the said fluorescence X-rays transmitted through said window.

The invention provides, in another broad aspect, apparatus for continuously presenting the solid component of a slurry for analysis comprising a cylindrical chamber having slurry inlet and outlet means, means for causing at least partial centrifugal separation of the solid component from the slurry, a window located in an end wall of the chamber so that in operation of the apparatus at least some of said solid component which has been separated from the slurry moves across the window in a continuous layer in contact therewith and slurry outlet means located axially in the chamber adjacent said window.

Preferably said outlet means comprise a conduit communicating with the interior of the chamber at an orifice provided centrally in a baffle member extending parallel to and spaced from the surface of the window. The spacing between the baffle member and the surface of the window is preferably adjustable.

The internal surface of the window may be coated, at least over the central portion thereof, with an abrasion resistant layer of synthetic resin material.

According to a preferred embodiment the means for causing centrifugal separation of the solid component, and said slurry inlet means, comprise an inlet pipe extending tangentially to the curved surface of the cylinder in a plane perpendicular to the axis thereof.

Preferably the window is formed of a synthetic resin film.

Preferably the window is supported in the chamber wall by a retaining member which is sealingly engaged with said wall and which has a film-engaging portion whereby movement of the retaining member relative to the chamber wall adjusts the tension in the film.

The preferred application of the apparatus referred to above is to the analysis of the solid component of a slurry by X-ray fluorescence. According, therefore, to another aspect of this invention, there is provided apparatus for testing continuously the concentration of a given element in the solid component of a slurry comprising apparatus for presenting the solid component for analysis as herein before defined in combination with analysing means for analysing radiation transmitted through the window. In a preferred embodiment, the apparatus includes means for inducing X-ray fluorescence of the solid component in said layer, said analysing means comprising means for analysing said X-ray fluorescence.

Preferably the analysing means is responsive to a predetermined component of the characteristic X-radiation of the given element.

Said means for inducing X-ray fluorescence are preferably located externally of said window and comprise an X-ray tube or a radioactive source of X-rays.

Means are preferably provided for adjusting the rate of flow of slurry through the chamber.

A purely exemplary description of the invention will be given with reference to the accompanying drawings, in which:

FIGURE 1 is a diagrammatic sectional view through apparatus for continuously presenting the solid component of a slurry for analysis constructed and arranged in accordance with the invention;

FIGURE 2 is a graphical representation of the effect of an X-ray fluorescence analysis of the concentration of the solid component in a slurry, and

FIGURE 3 is a diagrammatic view, partly in section, of X-ray fluorescence analysis apparatus employing the invention.

Referring to FIGURE 1 the apparatus comprises a cylindrical chamber **10** formed of duralumin which is closed at one end by a plane wall **11** and which is open at the other end. The chamber **10** is bounded at its open end by a continuous radially extending flange **12** having a bevelled surface **12***a* at the entrance to the chamber **10**.

The open end of the cylindrical chamber **10** is closed by a thin window **13** which extends over the flange **12** and is held in contact with the surface of the flange **12** by an annular duralumin retaining member **14**. The radially inner portion of the member **14** is formed with a continuous raised lip **15**, which is disposed parallel to the bevelled surface **12***a* of the flange **12**. The retaining member **14** is adjustable relatively towards or away from the flange **12** by means of screws **16** which pass through the member **14** and are received in threaded apertures in the flange **12**. The position of the retaining member **14** relative to the flange **12** determines the tension in the window **13**. The joint between the flange **12** and the retaining member **14** is rendered fluid tight by means of an annular rubber gasket **17**.

The chamber **10** is provided adjacent the end wall **11**

with a slurry inlet pipe **18** which extends tangentially to the cylindrical surface of the chamber **10** in a plane perpendicular to the axis thereof. Means (not shown) are provided for pumping slurry into the chamber through the inlet pipe **18** at an adjustable rate.

Slurry outlet means are provided in the form of an outlet pipe **19** extending axially of the cylindrical chamber **10** and terminating in the chamber **10** at an orifice **20** provided centrally of a disc-like plane baffle member **21**. The baffle member **21**, which in the illustrated embodiment is formed as a continuous flange at the end of the outlet pipe **19**, extends parallel to and is spaced a short distance from the surface of the window **13**. The outlet pipe **19** passes in screw-threaded engagement through a brass bush **22** which is fluid-tightly mounted in an aperture centrally of the end wall **11**. The spacing between the baffle member **21** and the surface of the window **13** is adjustable by screwing the outlet pipe **19** into or out of the chamber **10** as necessary.

The window **13** is formed of polyester film, such as that known by the trade name "Melinex," supported by a nylon mesh. The tension in the film is adjusted by suitable setting of the screws **16**. The thickness of the window varies according to the penetrating power of the characteristic X-rays being measured, a typical thickness being 0.005 inch. To protect the window **13** from abrasion by the slurry the central portion of the surface of the window **13** opposite the orifice **20** is provided with a protective layer **23** such as, for example, a layer 0.1 to 0.2 mm. thick of impact adhesive.

In operation of the apparatus, slurry entering the chamber **10** through the tangential inlet pipe **18** has imparted to it a vortex flow which induces at least partial centrifugal separation of the solid component from the slurry, said solid component being forced centrifugally against the cylindrical walls of the chamber **10**. The slurry flows between the walls of the chamber **10** and the baffle member **21**, leaving the chamber through the orifice **20** and the outlet pipe **19**. The said solid component which has been separated from the slurry passes continuously along a vortex path from the cylindrical wall of the chamber **10** radially inwardly towards the axis of the chamber **10** in a continuous layer in contact with the window **13**. As a result, the solid component of the slurry is presented to the window **13** as a continuous close packed layer, so that analysis of said solid component may be made independently of the remainder of the slurry and measurements can be made without having to take into account the concentration of the solid component in the slurry.

This is illustrated by the particular application of the invention to continuous "on stream" analysis of a slurry by the X-ray fluorescence technique. This technique, which is described, for example, in U.K.A.E.A. Research Group Report AERE–R4474, is applicable to the determination of the concentration of a given element in the solid component of a slurry. Such a determination has previously necessitated a knowledge of the concentration of the solid component in the slurry, which in many cases is difficult to obtain. If, however, the slurry is presented for analysis by passing it through apparatus such as that in FIGURE 1, then the layer of solid component which forms at the window **13** can be analysed independently of the remainder of the slurry.

The X-ray fluorescence analysis apparatus illustrated schematically in FIGURE 3 includes a slurry presenting chamber **10** in accordance with the invention. The slurry to be analysed is circulated through the chamber **10**, in the manner described above, in a closed circuit including a peristaltic pump **25** and a reservoir **26**. A stirrer **27** is disposed in the reservoir **26**.

A radioactive point source **30** (FIGURE 3) emitting X-rays is mounted externally of the window **13** of the presenter chamber **10**. The source **30** is so selected that the X-rays emitted thereby cause X-ray fluorescence of the solid component of the slurry in the layer behind the window **13**. The X-ray fluorescence passes out through the window **13** and is analysed using a detecting system. The detecting system, shown diagrammatically, comprises a scintillation counter **32** which is rendered responsive to a predetermined characteristic X-radiation of the given element by the interposition of suitable filters **33**. The intensity of the fluorescence is indicated by a scaler-time unit **34** connected to the scintillation counter **32** through an amplifier **35** and pulse height analyser **36**. The fluorescence intensity will be a function of the concentration of the given element in the solid component of the slurry, independently of the concentration of the solid component in the slurry, provided the depth of the layer formed at the window **13** exceeds a critical depth equal to the depth of the solid component from which the characteristic X-rays detected by the detecting system originate. The spacing between the baffle member **21** and the window **13** is adjusted to a value in accordance with the physical characteristics of the slurry under analysis.

FIGURE 2 illustrates graphically an analysis of a slurry the solid component of which contains barium. The variation of the fluorescent X-ray intensity of barium Kα characteristic radiation is plotted as a function of the concentration in a slurry of the solid component, using the apparatus for presenting the solid component according to the invention. The X-ray intensity is indicated as the count rate per second of a proportional counter which is responsive to the barium Kα radiation, the concentration of the solid component or "solid content" of the slurry being expressed as a percentage by weight. It is seen that, above about 1% solid component concentration, the intensity measured is substantially independent of the solid content of the slurry, illustrating the effectiveness of the apparatus in separating the solid component and presenting it for analysis.

We claim:

1. A method of testing continuously the concentration of a given element in the solid component of a slurry comprising the steps of:

partially centrifugally separating a solid component from the slurry;

moving the solid component so separated in a continuous layer across and in contact with a window; and

analyzing the solid component in said layer by means of radiation transmitted through said window.

2. A method as claimed in claim **1** wherein said step of analyzing includes the steps of:

inducing X-ray fluorescence of the solid component in said layer; and

analyzing the said fluorescence X-rays transmitted through said window.

3. Apparatus for continuously presenting the solid component of a slurry for analysis comprising a cylindrical chamber having slurry inlet means, means in said chamber for causing at least partial centrifugal separation of the solid component from the slurry, a window located in an end wall of the chamber and slurry outlet means located axially in the chamber adjacent said window, whereby in operation at least some of said solid component which has been separated from the slurry moves across the window in a continuous layer in contact therewith before leaving via the outlet means.

4. Apparatus as claimed in claim **3** wherein said outlet means comprise a conduit extending centrally into the chamber and a baffle member disposed parallel to and spaced from the surface of the window.

5. Apparatus as claimed in claim **4** including means for adjusting the spacing between the baffle member and the surface of the window.

6. Apparatus as claimed in claim **3** wherein the internal surface of the window is coated, at least over the central portion thereof, with an abrasion-resistant layer of synthetic resin material.

7. Apparatus as claimed in claim **3** wherein the means for causing centrifugal separation of the solid component,

and said slurry inlet means, comprise an inlet pipe extending tangentially to the curved surface of the cylindrical chamber in a plane perpendicular to the axis thereof.

8. Apparatus as claimed in claim **3** wherein the window is formed of a synthetic resin film.

9. Apparatus as claimed in claim **8** wherein a retaining member supports the window in the chamber wall, said retaining member being sealingly engaged with said wall and having a film-engaging portion whereby movement of the retaining member relative to the chamber wall adjusts the tension in the film.

10. Apparatus as claimed in claim **8** wherein said film is a polyester film.

11. Apparatus for testing continuously the concentration of a given element in the solid component of a slurry comprising apparatus for presenting the solid component for analysis as claimed in claim **3** in combination with analysing means for analysing radiation transmitted through the window.

12. Apparatus as claimed in claim **11** including means for inducing X-ray fluorescence of the solid component in said layer, said analysing means comprising means for analysing said X-ray fluorescence.

13. Apparatus as claimed in claim **12** wherein the analysing means is responsive to a predetermined component of the characteristic X-radiation of the given element.

14. Apparatus as claimed in claim **12** wherein the means for inducing X-ray fluorescence are located externally of said window and comprise a source of X-rays.

15. Apparatus as claimed in claim **11** including means for adjusting the rate of flow of slurry through the chamber.

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U.S. Cl. X.R.

250—43.5