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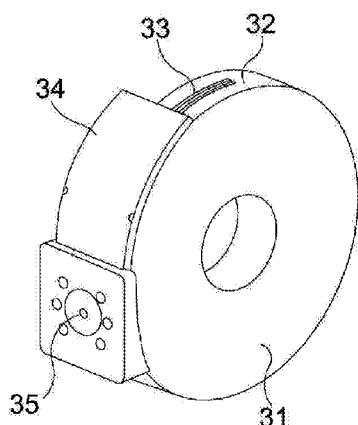


Figure 3

(57) Abstract: A method of manufacture of an article by continuous solid-state extrusion, the method comprising: providing a metallic particulate feedstock material; feeding the particulate feedstock material at a feed rate into a groove moving with a velocity; transporting the material in the groove to a diverter configured to divert at least a portion of the material into a die chamber port leading to at least one extrusion die; and extruding material through the extrusion die thereby producing the article; wherein the level of particulate material in the groove is controlled such that sufficient extrusion pressure is maintained without being such as to cause a failure of the diverter.



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Manufacture of Metal Articles

The present invention relates to the manufacture of metal articles, more particularly the manufacture of metal articles by extrusion. The invention also relates to the manufactured articles and the use of such articles.

Titanium is a highly desirable engineering material due to its high specific strength, good corrosion resistance and for certain applications, a low elastic modulus. However, despite high availability of its ores within the Earth's lithosphere, titanium has failed to gain wider acceptance, due to high extraction, purification and fabrication costs.

An ongoing drive for low-cost titanium products has focused on reducing the cost of extracting the metal from its ores. Processes such as the FFC Cambridge Process, CSIRO TiroTM and Cristal Metals Armstrong ProcessTM all produce titanium in a particulate form, which then needs further processing to create functional components.

Hence, there is a need for downstream processes to focus on reducing the costs of the final product so as to mitigate any further cost increases and, potentially, bring titanium into the commodity metal market. There is a further need for such downstream processes to be capable of handling titanium in a particulate form.

Titanium ingots currently require multiple refinement steps within a Vacuum Arc Remelt (VAR) furnace, grain breakdown (cogging) and reheats. The billet then undergoes numerous stages of thermomechanical processing designed to reduce the cross section and optimise the microstructure based on the required final product. Secondary processing, e.g. drawing or rolling, typically may be carried out to produce a long product such as a bar or wire or a flat product such as a sheet or slab. Finally machining and pickling steps may be performed to give the product the best geometrical tolerances and improve the surface finish. This process is complex, energy intensive and expensive. Such a conventional ingot metallurgy route typically may involve 40-50 processing steps and is a batch process.

Powder metallurgy could offer a possible solution to low cost downstream processing as it can form near-net shapes directly from particulate feedstock. Typically, methods such as Hot/Cold Isostatic Pressing (HIP/CIP), Extrusion and Direct Particle Rolling (DPR) are first required to consolidate the particulate material into a green billet/sheet with further finishing steps to improve density, surface quality and grain size. Inert atmospheres are usually required to prevent significant oxygen pick up

from the large surface area presented by the powder particles. Accordingly, these known methods typically are a batch process and may be complex, energy intensive and expensive.

5 An ideal powder metallurgy process should be continuous, require minimal powder pre-conditioning and be tolerant of changes in product cross section and size. However, to date a satisfactory titanium powder continuous consolidation/processing route has not been developed.

A first aspect of the invention provides a method of manufacture of an article by continuous solid-state extrusion, the method comprising:

- 10 providing a metallic particulate feedstock material;
feeding the particulate feedstock material at a feed rate into a groove moving with a velocity;
transporting the material in the groove to a diverter configured to divert at least a portion of the material into a die chamber port leading to at least one extrusion die; and
extruding material through the extrusion die thereby producing the article;
- 15 wherein the level of particulate material in the groove is controlled such that sufficient extrusion pressure is maintained without being such as to cause a failure of the diverter.

Advantageously, the invention may provide an improved processing route for a metallic particulate feedstock material.

- 20 Advantageously, sufficient plastic deformation may occur during the method of manufacture according to the invention to generate sufficient heat and pressure to bond individual metallic feedstock particles.

- 25 Typically, the particulate feedstock material may not require any pre-conditioning.

- Typically, friction between the groove and the particulate feedstock material and/or the amount of work applied to the material by the diverter may provide sufficient heat to consolidate and deform the material in the extrusion process. Accordingly, no pre-heating of the particulate feedstock material
- 30 may be required. Embodiments, in which the particulate feedstock material is not pre-heated prior to being fed into the groove may be particularly preferred. Beneficially, as a result of not having to pre-heat the particulate feedstock material, energy and cost savings may be realised. Additionally or alternatively, materials handling may be easier and/or safer.

- 35 In an embodiment, the particulate feedstock material may be fed into the groove at a temperature of no more than 100°C, no more than 50°C or no more than 40°C. The particulate feedstock material

may be fed into the groove at or around ambient temperature. Typically, ambient temperature may be from 15°C to 30°C, e.g. approximately 20°C.

Advantageously, when the particulate feedstock material is fed into the groove without any pre-heating, e.g. at or around ambient temperature, the invention typically need not be carried out in a protective (inert) atmosphere, e.g. argon. Conveniently, the invention may be carried out in air. Being able to carry out the invention in air, as opposed to in a protective (inert) atmosphere, may reduce apparatus cost and complexity and/or advantageously may allow the invention to be carried out safely in a wider range of locations and environments.

In an embodiment, no pre-heating of the apparatus or tool may be required. Friction between the particulate feedstock material and, for example, the groove and/or the diverter may assist in heating the apparatus or tool, in use.

However, in some embodiments, one or more parts of the apparatus or tool may be heated prior to the particulate feedstock material being fed into the groove and/or during carrying out of the method. Heating one or more parts of the apparatus or tool, e.g. the groove and/or the diverter, prior to feeding the particulate feedstock material into the groove may assist in initially consolidating the particulate feedstock material. The amount of heat supplied in addition to frictional heating may be varied while the method is being carried out.

In an embodiment, the level of particulate material in the groove may be controlled such that it does not exceed a predetermined level. The predetermined level may be selected so as to prevent the diverter from failing.

In an embodiment, controlling the level of particulate material in the groove may comprise controlling the feed rate and/or the velocity of the groove.

In an embodiment, a relief facet associated with the groove may be provided so as to provide an overflow for excess particulate material, thereby preventing, in use, the level of particulate material in the groove from exceeding a critical fill height.

In an embodiment, the extrusion die may be oriented at an angle to the groove.

The extrusion die may be oriented at an angle of 30 degrees or more to the groove, e.g. the extrusion die may be substantially perpendicular to the groove.

The groove may have any cross-sectional shape. In an embodiment, the groove may have a flat base, a stepped base, a sloping base or a curved base. For instance, the groove may be rectangular or semi-circular in cross-section.

- 5 In an embodiment, the method may comprise measuring a current to a motor causing the groove to move and, optionally, adjusting the feed rate and/or the velocity of the groove in response to the current measurement.

In an embodiment, the groove may be provided on a wheel.

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In an embodiment, the method may comprise measuring a torque on the wheel and, optionally, adjusting the feed rate and/or the velocity of the groove in response to the torque measurement.

In an embodiment, the wheel may be rotated at a speed of 5 RPM or more, e.g. 6 RPM or more.

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In an embodiment, the particulate feedstock material may comprise titanium, aluminium, copper, zirconium, niobium or tantalum or alloys thereof.

The particulate feedstock material may be regularly or irregularly shaped.

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If the particles of the particulate feedstock material are too big, then the particles may not be sheared at the diverter. If the particles of the particulate feedstock material are too small, then they may not flow smoothly when being fed into the groove.

- 25 In an embodiment, the particulate feedstock material may have an average particle size of up to 2 mm, up to 1 mm, up to 500 μm or up to 300 μm . The particulate feedstock material may have an average particle size of at least 10 μm , at least 20 μm , at least 30 μm or at least 40 μm . In an embodiment, the particulate feedstock material may have an average particle size of at least 45 μm and/or up to 150 μm .

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In an embodiment, the particulate feedstock material may comprise machining swarf from single or mixed sources.

- 35 The particulate feedstock material may comprise recycled or reclaimed material, e.g. swarf, turnings and/or cuttings. The particulate feedstock material may be produced at least in part from one or more components which have reached the end of their service life.

In an embodiment, the method may comprise the preliminary step of producing the particulate feedstock material.

The diverter may comprise an abutment, which may be received at least partially within the groove.

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The predetermined level may depend on the characteristics of the particulate feedstock material and/or on the set-up, dimensions and geometry of the apparatus used. For instance, the predetermined level may depend at least in part on the shape and dimensions of the groove. For a particulate feedstock material comprising a titanium or titanium alloy powder, the predetermined level may be from 20 mm to 50 mm.

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In an embodiment, in which the particulate feedstock material comprises CP-Ti HDH powder with a particle size fraction of 45 μm to 150 μm is continuously extruded using a Conform 315i machine, the predetermined level may be at least 25 mm and/or up to 45 mm, preferably be at least or up to 30 mm and/or at least or up to 35 mm.

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In an embodiment, the article may comprise a rod or a wire or strip. The article may have a profile of any shape, e.g. round, elliptical, triangular, rectangular, pentagonal, hexagonal, heptagonal, octagonal etc. Alternatively, the article may have a more complex profile, e.g. an L, T, U, I, X, W or tube profile.

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A second aspect of the invention provides a method of manufacture of a product comprising: providing an article manufactured according to the first aspect of the invention; and subsequently processing the article to form the product.

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In an embodiment, subsequently processing the article may comprise: mechanically deforming the article, e.g. by drawing; heat treating, e.g. annealing, the article; machining the article; and/or combining, e.g. joining, the article with one or more other components; and/or surface treatment, e.g. shot peening.

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In an embodiment, the product may comprise a spring. Alternatively, the product may comprise: a bolt, a rivet or other fixing structure; a wire product such as welding wire, dental wire, spring wire forms; an engine valve; a connecting rod; or part of a structure such as a building or a vehicle, e.g. an automobile (such as a car, a lorry or a bus) or an aircraft.

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A further aspect of the invention provides an apparatus for manufacture of an article by continuous solid-state extrusion of a particulate feedstock material comprising a movable groove, the movable

groove being operable to transfer feedstock material to a diverter configured to divert at least a portion of the material into a die chamber port leading to at least one extrusion die, wherein the apparatus further comprises a relief facet configured to receive excess material from the groove to ensure that the level of material within the groove does not exceed a predetermined amount.

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In an embodiment, the apparatus may comprise a motor operable to move the groove at a velocity.

In an embodiment, the apparatus may comprise a feed device configured to feed the particulate feedstock material into the movable groove.

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In an embodiment, the groove may be provided on a wheel.

In an embodiment, the groove may be bounded by a stationary shoe along at least a portion of its path to the diverter, e.g. between the or a feed device and the diverter.

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In an embodiment, the shoe may be configured to provide a decreasing groove cross-section as the groove transports the material towards the diverter. Alternatively, the shoe may be configured to provide an increasing groove cross-section as the groove transports the material towards the diverter. Alternatively, the shoe may be configured to provide a substantially uniform cross-section as the groove transports the material towards the diverter.

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The diverter may comprise an abutment, which may be received at least partially within the groove.

A further aspect of the invention provides a method of controlling a solid-state continuous extrusion apparatus, the apparatus comprising a movable groove for transporting, in use, particulate feed material, to a diverter and an electric motor operable to cause the groove to move, the method comprising measuring a current to the motor.

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Optionally, the method may comprise the step of varying, in response to measuring the current to the motor, the speed of movement of the groove and/or the rate of feeding the particulate feed material into the groove.

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Optionally, the method may comprise comparing the measured current with a reference value.

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A further aspect of the invention provides a method of controlling a solid-state continuous extrusion apparatus, the apparatus comprising a rotatable wheel with a groove for transporting, in use,

particulate feed material, to a diverter and a motor operable to cause the wheel to rotate, the method comprising measuring a torque on the wheel.

5 Optionally, the method may comprise the step of varying, in response to measuring the torque on the wheel, the speed of rotation of the wheel and/or the rate of feeding the particulate feed material into the groove.

Optionally, the method may comprise comparing the measured torque with a reference value.

10 Optionally, the method may further comprise measuring the current to the motor.

An advantage of the present invention is that it may enable the solid-state, continuous processing of a metallic powder, e.g. titanium, into a wire or a rod. A product such as a spring may then be made from the wire or rod.

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Advantageously, the invention may provide for economical and/or convenient solid-state, continuous processing of a metallic particulate feedstock (e.g. a powder), which may, in particular, comprise titanium or a titanium alloy such as Ti-6Al-4V or Ti-6Al-7Nb.

20 In an embodiment, the metallic particulate feedstock may comprise an alpha-rich titanium alloy with a molybdenum equivalent (Mo.eq.) from 0-6.

A further aspect of the invention provides a continuously extruded wire, rod or strip, typically comprising titanium or a titanium alloy, wherein the wire, rod or strip has, in cross-section, a
25 microstructure comprising a bi-modal distribution of fine-grained regions and coarse-grained regions with respect to the flow pattern.

A further aspect of the invention provides a product or component comprising a continuously extruded wire, rod or strip, typically comprising titanium or a titanium alloy, wherein the wire, rod or
30 strip has, in cross-section, a microstructure comprising a bi-modal distribution of fine-grained regions and coarse-grained regions with respect to the flow pattern.

The product or component may comprise a spring. Alternatively, the product or component may comprise: a bolt, a rivet or other fixing structure; a wire product such as welding wire, dental wire,
35 spring wire forms; an engine valve; a connecting rod; or part of a structure such as a building or a vehicle, e.g. an automobile (such as a car, a lorry or a bus) or an aircraft.

Advantageously, this invention may offer a one-step, continuous, downstream processing route for the current low-cost titanium powders, e.g. commercially pure (CP) grade 2 hydride-dehydride (HDH) titanium powder, produced by companies such as Metalysis, Cristal Metals and CSIRO. Rapid fabrication of such low-cost titanium powders to final product utilizing the present invention may offer a step change in the economics of titanium. This change could result in titanium being used for applications such as in the automotive market where up until now it has been priced out by steels.

Other metallic particulate feedstocks may also be processed in accordance with the invention. For instance, the invention may be utilised to process a particulate feedstock comprising aluminium, copper, zirconium, niobium or tantalum, and alloys thereof.

A further aspect of the invention provides an automobile, e.g. a car, lorry or a bus, comprising one or more components, e.g. springs, according to any aspect of the invention.

A further aspect of the invention provides an aircraft comprising one or more components, e.g. landing gear springs, according to any aspect of the invention.

In order that the invention may be well understood, it will now be described by way of example only with reference to the accompanying drawings in which:

Figure 1 shows a cross-section of a generic Conform rotary extrusion machine;

Figure 2 illustrates the shear created in a powder in a flash gap by a moving Conform wheel;

Figure 3 shows an overview of a simulation tooling setup;

Figure 4 shows a section through the simulation tooling setup showing a partially extruded workpiece;

Figure 5 shows a die back end discard;

Figure 6 shows the placement of truncated workpieces for FEM analysis of the effective abutment stress and grip length relationship;

Figure 7 shows an abutment and die port following an interrupted test;

Figure 8 shows the fracture surface of a failed abutment with partially consolidated titanium processed over the top;

Figure 9 shows a die chamber at the end of an experimental trial;

Figure 10 shows a die chamber and entry block within the shoe at the end of a trial showing good consolidation;

Figure 11 shows a die back end discard;

Figure 12 shows a die back end discard;

Figure 13 is a graph of abutment stress as a function of wheel speed for rotary extrusion of commercially pure titanium powder;

Figure 14 is a graph of abutment stress as a function of wheel speed for rotary extrusion of Ti-6Al-4V powder;

Figure 15 is a graph of abutment stress as a function of wheel speed for rotary extrusion of commercially pure titanium powder;

- 5 Figures 16A, 16B and 16C are graphs of abutment stress as a function of wheel speed for rotary extrusion of commercially pure titanium powder;

Figure 17 is a graph of shear ratio plotted for different wheel speeds down the abutment;

Figure 18 is a macro image of the flow within a cross-section of extruded product taken using polarised light;

- 10 Figure 19 is a polarised light micrograph of the cross-section of extruded product shown in Figure 18;

Figure 20 is a composite macrograph of a section of a die discard taken using polarised light;

Figure 21 is a simulated strain distribution profile within a Conform machine;

Figure 22 is a simulated strain rate distribution profile within a Conform machine;

Figure 23 is a simulated stress field within a Conform machine;

- 15 Figure 24 is a simulated temperature distribution within a Conform machine;

Figure 25 is a simulated velocity distribution within a Conform machine;

Figure 26 shows flow lines of tracked points through the abutment zone with dead zones indicated;

Figure 27 is a graph of effective abutment stress calculated from torque measured on the wheel in truncated finite element simulations;

- 20 Figure 28A is a graph of average abutment stress over sample simulations plotted against wheel groove fill depth;

Figure 28B is a graph of average abutment stress over sample simulations plotted against grip length for four different metallic powders;

Figure 29 is a Conform machine data graph from an experimental trial;

- 25 Figure 30 is a Conform machine data graph from another experimental trial;

Figure 31 is a Conform machine data graph from another experimental trial;

Figure 32A is a Conform machine data graph for an initial portion of another experimental trial;

Figure 32B is a Conform machine data graph for a later portion of the same experimental trial as Figure 32A;

- 30 Figure 33 shows the grain size distribution in a section of titanium rod manufactured in accordance with the invention;

Figure 34 show a typically grain size distribution in a cross-section of an aluminium rod manufactured by continuous extrusion;

- 35 Figure 35 shows the grain structure within two different regions of a section of titanium rod manufactured in accordance with the invention; and

Figure 36 shows the macrostructure of a section of titanium rod manufactured in accordance with the invention.

The Conform rotary extrusion process was invented in 1972 by Derek Green at the Springfields Laboratories, UK Atomic Atomic Energy Authority. While the Conform of copper and aluminium rod has been investigated since the invention of the process, very little has been published on the Conform of particulate products. In particular there is a gap in the literature and in industry about the continuous extrusion of titanium.

The Conform process was initially conceived as a method for continuously extruding electrical grade aluminium and copper alloys. Its potential for powder processing was also mentioned in US3765216. US4552520 described modifications to the original machine to improve the process for powder feed including the addition of a hopper.

Since the initial work there has been little published information on the behaviour of particulate feed in the Conform process and in particular the properties of the resultant product. D. J. Marsh (The Extrusion of Copper by the Conform Continuous Extrusion Process, Tech. rep., Reactor Fuel Element Laboratories (1977)) described some developmental work performed with copper powder as well as chopped copper electrical cables and found that it was possible to process a wide range of particulate morphologies with no detrimental effects to the product mechanical properties. Pardoe ("Conform" Continuous Extrusion of Metal Powders into Products for Electrical Industry: Development Experience, Powder Metallurgy 22 (1) (1979) 22-28) focused purely on processing particulate aluminium and copper feedstocks of various size fractions, morphologies and chemistries. While it was mentioned that there were variations in visual product quality and mechanical properties very little was mentioned about the process parameters used in the experiments. There was also no discussion of the microstructures or textures evolved. The methods described are incomplete and don't allow replication or application to titanium.

Etherington (Conform and the Recycling of Non-Ferrous Scrap Metals, Conservation and Recycling 2 (March) (1978) 19-29) described work in which Dryflo copper was consolidated using an experimental Conform machine. This work showed the internal macro-structure of the extrudate, which was revealed to be highly heterogeneous with flow lines delineated by entrained oxides.

Accordingly, it will be appreciated that there is a fundamental lack of understanding of the behaviour of the particulate metallic feedstock in a continuous rotary extrusion process such as the Conform process. Without this understanding, it will not be possible to improve such processes for aluminium or copper feedstocks or even to apply such processes to other metals, in particular particulate feedstocks comprising titanium, zirconium, niobium or tantalum, and alloys thereof.

Figure 1 shows a cross section of a generic Conform rotary extrusion machine 1. The machine 1 comprises a grooved wheel 2, which is used to drive particulate feedstock from a feeding apparatus or a continuous length of rod feedstock around the wheel 2 towards an abutment 4, which protrudes into the groove. The diameter of rod feedstock may be chosen to match the wheel and groove dimensions for the desired final product. As illustrated in Figure 1, the feeding apparatus may comprise a hopper 3, which may be funnel-shaped, and a vibrating chute 8. The vibrating chute 8 is located between the hopper 3 and the grooved wheel 2. The chute 8 need not be vibrating. Alternatively, the machine 1 may not comprise a chute, in which case particulate feedstock may pass directly from the hopper 3 into the groove.

The groove is bounded by an enclosing stationary shoe 5 positioned around a portion (approximately 1/6 in the illustrated example) of the circumference of the wheel 2, which is configured such that, in use, the passing feedstock experiences an ever reducing groove cross section. When a workpiece (i.e. the material within the groove) reaches the abutment 4, the workpiece is upset and diverted into a die chamber port 6. In the example shown in Figure 1, the die chamber port 6 extends radially outwards from the wheel 2. The die chamber port 6 communicates with a die 7. As material fills the die chamber port 6, pressure builds up behind the die 7. The large amount of work imparted to the workpiece causes it to heat up rapidly, decreasing its flow stress and reducing the required extrusion pressure. The workpiece is continuously extruded through the die 7 once the required pressure is developed within the material and exits the machine 1 as extruded article 10. Providing the die profile chosen is suitable for both the machine capabilities and material properties of the feedstock, the extrudate (the extruded article 10) typically exits the machine with a bright surface finish and can be quenched immediately, e.g. with an inline water trough (not shown).

During operation of the machine 1, the temperature and stress profile within the feedstock reaches a steady state where predictable product properties can be established. Typically, a certain quantity of the workpiece is lost in the form of flash through the gap between the abutment 4 and the wheel 2. This flash may coat a portion of the abutment 4, e.g. an outside heel groove, and may be scraped off after use of the machine 1. The groove may also be coated in flash, which may remain on the wheel 2 and may be reintroduced back into the process with each rotation. As illustrated in Figure 1, the machine 1 may comprise a scraper 9 configured to scrape at least a portion of any remnant flash off the wheel 2.

Typically, the deformation of the metal within the Conform machine is impossible to view when the machine is running due to the abutment zone being obscured by the wheel and shoe arrangement. Temperatures may be measured by thermocouples positioned at the back faces of the abutment. However, at this location, the thermocouples may fail to react quickly to changes within the process.

Such measurements may also fail to show the temperature distribution within the feedstock, which may enter the machine at room temperature and can be heated to in excess of 500 °C within a few seconds.

- 5 There are many different tooling setups due to the different size of Conform machines available and possible product cross section but all have the same basic bulk deformation route before true extrusion can begin.

- 10 An advantage of the present invention is that no pre-heating of the feedstock is typically required. A further advantage is that the invention can be carried out without the need for a protective (inert) atmosphere.

- 15 In the applicant's experiments, rod was continuously extruded from commercially pure grade 2 titanium hydride-dehydride powder (CP-Ti HDH) using a Conform machine. The applicant's initial experiments conducted to produce this rod were not rigorous enough to link physical properties to the process parameters with a high degree of certainty. In order to make such a link and develop a working model to predict microstructure and mechanical properties from the rod further experiments were performed.

- 20 For instance, a section of rod was chosen based on homogeneity of microstructure and mechanical properties and was subjected to a wire drawing study where a suitable processing schedule was determined.

- 25 In these further experiments, a two-stage approach was taken to developing an understanding of titanium within the Conform rotary extrusion process. Firstly, three separate attempts were made to extrude rod from the same machine as before. Two attempts were made using a rectangular-profiled die and StelliteTM 21 abutment and one with a 5-mm round profiled die with a W-25 w.% Re abutment chosen because of its superior hot working strength. All three attempts successfully extruded a short rod. However, none of the attempts produced more than 100 mm of rod/strip due to premature failure
30 of the abutment. Process measurements made by the Conform machine indicated that the torque generated by the wheel during the failure of the abutments was significantly higher than during the preceding successful extrusion. The wheel speed during the start-up phase, when fresh cold powder is fed into an empty machine, was also inconsistent between tests. While most operation parameters were measured, the powder feed rate into the machine was impossible to both fully control and to
35 reproduce reliably between tests.

Powders of pure aluminium, high conductivity oxygen free copper and AgSnO_2 have been reported to be insensitive to both powder feed rate and wheel speed. However, the applicant discovered that this is not the case for CP-Ti HDH powder. This discovery necessitated further work to develop both a suitable and reproducible processing window for CP-Ti HDH powder, which could then be adapted for other higher strength titanium alloy powders.

The experimental trials were carried out on a Conform 315i machine with a range of different tooling setups to determine what effect certain process parameters would have on the final product. The die dimensions and abutment materials used in the experimental trials are summarized in Table 1 below.

StelliteTM 21 is a corrosion resistant CoCr alloy, which comprises a CoCrMo alloy matrix containing dispersed hard carbides.

Trial	Die	Abutment
2	5 mm round	Stellite TM 21
3.1	6.63 x 2.24 mm rectangular (Horizontal)	Stellite TM 21
3.2	6.63 x 2.24 mm rectangular (Vertical)	Stellite TM 21
4	5 mm round	W-25 w.% Re

Table 1: Experimental Trials Tooling

In the experimental trials, the die, die chamber port and abutment were preheated to 500 °C and held for 10 minutes to ensure thermal homogeneity. The wheel was started at a nominal speed of 2 RPM and allowed to rotate for a number of turns for the abutment to clear any protrusions from any previous material left in the groove. Unprepared CP-Ti Grade 2 HDH powder (45-150 μm) was poured onto a vibrating folded plate, which fed the powder directly into the wheel groove during the machine's operation. As the powder was fed in by a hand scoop the feed rate was not consistent or measurable. The wheel speed was raised by 2 RPM when it was felt that the powder had begun to consolidate above the abutment. On occasions when loose powder ignited in the bottom of the machine prior to consolidation occurring, the wheel had to be stopped to allow the fire to burn itself out.

Conform machines have the capability to display recorded data from various sensors during operation. This data can be used for the real-time monitoring of the process or exported later for post-operation analysis. An important part of the applicant's experimental work was the analysis of these files to determine certain stages of the process and to recognise how the process develops throughout the machine's operation. Raw data relating to metrics such as wheel speed, motor current, tooling temperatures and extrusion velocities were produced and analysed.

Effective Abutment Stress

From the data produced by the Conform machine after runtime it was possible to calculate the torque produced by the wheel in order to maintain the user-set wheel speed. This torque reflects the amount of work going into the powder and can give an indication of the stress levels on the abutment. The stress within the abutment was found to be near critical levels throughout the processing of commercially pure titanium powder, which has a yield strength two to three times greater than any other material (e.g. copper or aluminium) that the machine tooling was designed for. Three of the four trials conducted in this study all resulted in a premature fracture of the abutment tip and subsequently increased the effective flash gap. While a certain flash gap is required to prevent the abutment interfering with the wheel groove if it is too large, then inadequate pressure will be evolved within the powder for consolidation and extrusion to occur.

The wheel torque is related to the ratio of the motor current and rotational speed through equation 1 below.

$$\tau = \frac{2\pi VI\gamma}{60\omega}$$

Equation 1

where V is the supply voltage of the DC motor driving the wheel, I is the operating current of the motor, γ is the motor's electrical to mechanical efficiency, assumed to be 0.8 and ω is the angular velocity of the wheel in rad s^{-1} . If this torque τ is considered to be a force working at a distance equal to the radius of the centre of the wheel groove R it is possible to determine an average stress acting on the abutment face (A_{abut}) within the groove through equation 2.

$$\tau = \frac{2\pi VI\gamma}{60\omega R A_{\text{abut}}}$$

Equation 2

This is a very simplified consideration of the situation and fails to account for varying grip lengths and changes in frictional coefficients. It did however yield a useful metric from which further work could be done to determine safe operating parameters for processing titanium powder within the Conform machine. The torque generated at the top and bottom of the groove will also be different resulting in a graduated stress distribution across the abutment face.

Powder Shear at the Wheel Feedstock Interface

When starting up the Conform process with powder feedstock the wheel groove is initially clean with a flash tolerance. It is necessary to coat the groove with processed powder during a startup phase to

contain further powder being fed in behind it. This has the result of heating up the fresh cold powder and reducing loose powder waste out of the groove. With powders of aluminium and copper it is possible to coat the wheel at low wheel speeds. However, previous experience with titanium HDH powder has indicated that there is a minimum wheel speed required to begin consolidation with such a higher strength powders.

As powder is initially fed into the groove from above the machine and reaches the abutment it has a bulk density less than that of its tap density. Particles are able to free-fall through the flash gap between the wheel groove and the abutment tip. There is an effective shear created between the surface velocity of the wheel and the stationary abutment tip. If there is enough powder to fill the flash gap up to the powder tap density and there is sufficient particle interlocking it is possible for the transfer of force between the wheel and the abutment using the powder as a medium. If there is no interaction between the powder particles and the abutment or groove there will be no shear within the powder bulk in the flash gap. With a filled abutment zone and a moving wheel there is a shear within the powder in the flash gap.

By way of illustration, Figure 2 helps to illustrate the concept of shear within the powder at the abutment tip. Figure 2 shows a circular region 21 positioned in the flash gap between an abutment 23 and a rotating wheel 22. The wheel is rotating in the direction indicated by arrow 24. The circular region 21 represents a region of interest within the powder that lends itself to the descriptive model of the theory of powder shear. The smaller arrows 25 (only one of which is labelled for clarity) indicate the relative velocity of powder particles at those points. Thus, for instance, powder particles adjacent the wheel 22 move at a higher velocity than powder particles adjacent the abutment 23.

Consider a single powder particle positioned in the middle of the flash gap at the height of the abutment top face. If it is released from this stationary point it will accelerate downwards under the action of gravity until stopped or deflected by another object. Now, consider the particle as a perfect sphere. When this particle is released it will experience a moment around its centre of mass dictated by the tangential force acting on it by the wheel and the reaction from the stationary abutment. This may not be a realistic consideration, as bulk powder will not act like a perfect sphere but it illustrates the forces acting on a section of powder within the flash gap. If the wheel 22 is stationary then the powder will flow through the flash gap only affected by its flow characteristics, which typically are dictated by the particle size distribution, particle morphology and tool wall friction. Typically, the flash gap may not be constant with drop from the abutment face and, typically may increase in size. Such an increase results in a diverging powder flow through the flash gap. After the powder flows past the initial gap it will experience an ever decreasing interaction with neighbouring particles until it is completely free of this interaction. The height at which this occurs below the abutment face will

vary for different powders and tool set-ups. For instance, wall friction may play a significant part in how much powder can be safely assumed to be in free fall. In addition, if the surface velocity of the wheel were less than the velocity the powder could attain under free-fall then the friction between the powder and the groove would prevent such free fall. On the other hand, if the wheel's surface velocity exceeded this minimum requirement then there would be an induced shear within the powder.

Consider now that the wheel is moving with a certain angular velocity ω . At a radius of the wheel groove r (for instance, r may be 150 mm), the tangential velocity is given by $v_t = \omega r$. The powder free fall velocity is given by the suvat equation $v = \sqrt{2gh}$, where the acceleration due to gravity is $g = 9.81 \text{ ms}^{-2}$ and h is the drop in height below the abutment face. As we assume that the powder is in unobstructed vertical freefall the vertical component of the wheel's tangential velocity is given by $v_y = v_t \sin(\theta)$. While this assumption is inaccurate it represents an upper-bound on the possible vertical velocity of the powder and hence lower limit on what the shear ratio will be at any point down the abutment. At each point in height from the abutment face it is possible to calculate the effective shear ratio between the wheel and abutment; the results of which are presented in section 3.2 figure 16. The effective shear within the powder will actually depend on many other factors. Particle size and morphology will affect the flow behaviour and hence likelihood of particle bridging across the flash gap. If this happens, the bridge will undergo a mixed shear and compression forcing powder consolidation higher up the wheel groove.

DEFORM 3D Finite Element Modelling of Conform

In addition to the experimental trials using the Conform machine, finite element modelling (FEM) was performed using the commercial DEFORMTM-3D software package and the built in flow stress model for grade 2 commercially pure titanium. FEM code is unable to cope with particulate feeds without modifications to create a hybrid discrete/finite element simulation, which is extremely computationally intensive when modelling the 10^6 - 10^7 particles required for a groove of a Conform machine of radius 6.5 mm and length 160 mm. Adaptations were made to a solid rod fed simulations to help understand the process with powder. Tooling CAD files were obtained from BWE Ltd, a manufacturer of Conform machines, to ensure dimensional accuracy. Figure 3 shows the 3D simulation setup before extrusion has begun. The workpiece (the consolidated material within the groove) was created by performing a boolean subtraction of an oversized geometry by the wheel and other objects to ensure maximum grip within the groove. A purely plastic flow stress model was chosen to reflect the primary deformation expected in a severe plastic deformation (SPD) process such as Conform.

Table 2 shows the relevant parameters used in the simulation. Friction coefficients (μ) were taken from the published literature, however the wheel-workpiece friction had to be increased to $\mu = 0.98$ to

ensure enough grip to develop the extrusion pressure. A constant wheel speed of 6 RPM was chosen as it represented a balance between the suboptimal startup speeds of 2-4 RPM and the successful 10 RPM steady state speed from the experimental trials. The simulation was allowed to run without a defined end point to ensure that thermal stability was attained. This occurred within a simulated real world time of 5 seconds.

Parameter	Value
Workpiece material	Grade 2 Titanium
Wheel speed	6 RPM / 0.628 rad s^{-1}
Friction	0.98 wheel 0.4 all other tools
Time step	0.005 s^{-1}
Convergence Δv	0.001
ΔF	0.01
<u>Initial Temperatures</u>	
Environment	20 °C
Workpiece	20 °C
Wheel	120 °C
All other tools	500 °C
<u>Geometries</u>	
Wheel radius	157.5 mm
Groove radius	6.5 mm
Circular die radius	2.5 mm
Flash gap	1.2 mm
Workpiece radius	13.0 mm

Table 2

- 10 The FE mesh was generated automatically by the program and the elements were refined in size around the die and abutment where the largest changes in workpiece geometry were expected and where the main deformation zone had previously been observed in other studies.

Figure 3 shows an overview of the simulation tooling set-up. A workpiece 33 is prefitted into a groove 32 in a wheel 31. A shoe 34 encloses a proportion of the circumference of the wheel. A die 35 through which the workpiece 33 will be extruded is also shown.

Figure 4 shows a cross-section of the simulation tooling set-up shown in Figure 3. In Figure 4, the workpiece 33 is shown as partially extruded. An abutment 36 is shown, which is configured to divert and deform the workpiece 33 into a die chamber port in communication with the die 35. A flash 37

from the workpiece 33 is shown between extending through gap between the wheel 31 and the abutment 36.

Die back-end discards from the experimental trials were analysed. Figure 5 shows an example of a die back-end discard 51. The die back-end discard 51 comprises a first face 52 corresponding to the wheel, a second face 53 corresponding to the abutment and a third face 54 corresponding to the die. Based on the applicant's analysis, the height above the abutment indicated by the double-headed arrow 55 can be assumed to be 100% dense. In this analysis, material above this region 55 within the wheel groove was not investigated to determine the possibility and nature of a density gradient within the powder. However, without wishing to be bound by any theory, this material is expected to have undergone some degree of compaction and hence may produce a measurable change in the torque on the wheel. In the experimental trials, there was a maximum fill height above the abutment of approximately 150 mm due to the geometry of the shoe entry block.

In order to investigate what effect the height of fully consolidated material has on the torque requirements of the wheel, six simulations were run with truncated workpieces of 7 mm, 13 mm, 18 mm, 24 mm, 30 mm and 40 mm in height.

Figure 6 shows schematically a Conform apparatus comprising a rotatable grooved wheel 61, a stationary shoe 62 enclosing a portion of the circumference of the wheel 61 and a die chamber port 63 located at an end of the shoe 62. The die chamber port 63 leads to a die 64, through which the material is extruded. An abutment 65, extending into the groove on the wheel 61 is configured to deform and divert the workpiece material, in use, into the die chamber port 63. There is a flash gap 66 between the wheel 61 and the abutment 65. In Figure 6, the heights of the six truncated workpieces are indicated by dotted lines and arrows with reference to the schematic Conform apparatus.

For the purposes of these simulations, the lowest four heights (7 mm, 13 mm, 18 mm, 24 mm) were selected as they represented certain parts of the die chamber geometry as shown in Figure 6. The tallest two (30 mm, 40 mm) were taken at regular intervals up the wheel groove.

In these simulations, the workpiece and wheel interface was assigned a sticking condition to ensure that there was a consistent grip. The workpieces all began the simulations at a uniform temperature of 20 °C while all other parameters were the same as the full workpiece simulations illustrated in Table 2. The wheel torque was extracted directly during post-processing and converted to an effective abutment stress using equation 2, which could then be compared with data from the full simulations

and from the experimental trials. Using this method it was possible to estimate the effective grip length of fully consolidated material within the Conform machine during experiments.

There are some limitations to using fully dense workpieces in the FE simulations. First, without wishing to be bound by any theory, it is unlikely that samples that are lower than the top of the die chamber port would ever be fully consolidated as there wouldn't be enough powder to generate the required pressure. Secondly, in order to generate the tallest samples during experiments excess powder would need to be added resulting in a layer of partially compacted powder above the fully consolidated material. At present, it is unknown what effect this partially compacted powder layer would have on the measured wheel torque and if it would even be significant when compared with the torque required for the fully dense layer.

Results

Experimental Trials

Trial 2 was carried out using a 5 mm round die. The abutment was made from Stellite™ 21. 16.7 mm of titanium rod product was extruded.

The Conform machine data for Trial 2 are shown in Figure 29. Wheel speed, measured in revolutions per minute (RPM), is indicated by a red line 291. Abutment temperature, measured in °C, is indicated by a green line 292. Wheel temperature, measured in °C, is indicated by a blue line 293. Abutment stress, measured in MPa, is indicated by the individual round data points 294 (for clarity, only one data point 294 is labelled).

Trial 3.1 was carried out using a 6.63 mm by 2.24 mm horizontally-oriented rectangular die. The abutment was made from Stellite™ 21. 5 mm of titanium product was extruded.

Figure 7 shows a close-up view of an abutment 71 and a die chamber port 72 following an interrupted test in Trial 3.1 with a wheel speed of 2 RPM. A material discard 73 from the back of the die can also be seen in Figure 7.

Figure 8 shows a close-up view of the die chamber port 72 at the end of Trial 3.1. A fracture surface 81 of the failed abutment can be seen with partially consolidated titanium processed over the top.

Analysis of material discards from the back of the die from each of the trials (Trials 2, 3.1, 3.2 and 4) showed that there appeared to be a self-regulating mechanism in action. Trial 3.1 included an

interrupted test to determine how the powder acted under the initial start-up conditions. From this interrupted test, it was determined that a wheel speed of 2-3 RPM was insufficient to begin consolidation. The second part of Trial 3.1 demonstrated partial consolidation of the powder and resulted in a discard, e.g. as shown in Figures 8 and 11. The abutment fractured before the discard was removed and resulted in a sheared back end to the sample. The overall shape of the discard shows the interface between consolidated and non-consolidated material early in the process.

The Conform machine data for Trial 3.1 are shown in Figure 30. Wheel speed, measured in revolutions per minute (RPM), is indicated by a red line 301. Abutment temperature, measured in °C, is indicated by a green line 302. Wheel temperature, measured in °C, is indicated by a blue line 303. Abutment stress, measured in MPa, is indicated by the individual round data points 304 (for clarity, only one data point 304 is labelled).

The effective abutment stress shown in the machine data in Figure 30 is extremely high during the early stages at a wheel speed of 2 RPM. This drops significantly when the wheel speed is doubled to 4 RPM but still resulted in the abutment failing. At present, it is unknown at which point during the second phase of this test the abutment failed.

Trial 3.2 was carried out using a 6.63 mm by 2.24 mm vertically-oriented rectangular die. The abutment was made from StelliteTM 21. 100 mm of titanium product was extruded.

Figure 9 shows a close-up view of a die chamber port 91 at the end of Trial 3.2 following a maximum wheel speed of 7 RPM.

Trial 3.2 demonstrated better consolidation and resulted in a 100 mm-length of extruded product being produced.

Figure 11 shows a first example of a die back-end discard 111 from Trial 3.2. The top 50-60 mm of the back-end discard 111 was extremely friable indicating that consolidation had not occurred in this region and that it was loose powder that had overfilled the groove. A yellow coloured oxide indicated that a lower temperature of 500-550 °C was obtained in this upper section. The lower 100 mm of discard was coloured purple and blue indicating much higher temperatures of up to 700-800 °C at the wheel interface.

Figure 12 shows a second example of a die back-end discard 112 from Trial 3.2. The die back-end discard 112 shown in figure 12 was tightly welded to the tools and had to be removed using a

hydraulic press resulting in cracking to the back of the sample. As a result it was not possible to make comments on exactly how much material was fully consolidated during this trial.

The Conform machine data for Trial 3.2 are shown in Figure 31. Wheel speed, measured in revolutions per minute (RPM), is indicated by red line 311. Abutment temperature, measured in °C, is indicated by a green line 312. Wheel temperature, measured in °C, is indicated by a blue line 313. Abutment stress, measured in MPa, is indicated by the individual round data points 314 (for clarity, only one data point 314 is labelled).

Trial 4 was carried out using a 5 mm round die. The abutment was made from W-25wt% Re. 100 mm of titanium product was extruded.

Figure 10 shows a die chamber port 101 and entry block within the shoe at the end of Trial 4. Good consolidation can be seen. The fractured abutment is hidden by a layer of titanium 102 processed over the top of it.

The Conform machine data for Trial 4 are shown, for an initial portion of the trial, in Figure 32A and, for a later portion of the trial, in Figure 32B.

In Figure 32A, wheel speed, measured in revolutions per minute (RPM), is indicated by a red line 321a. Abutment temperature, measured in °C, is indicated by a green line 322a. Wheel temperature, measured in °C, is indicated by a blue line 323a. Abutment stress, measured in MPa, is indicated by the individual round data points 324a (for clarity, only one data point 324a is labelled).

In Figure 32B, wheel speed, measured in revolutions per minute (RPM), is indicated by a red line 321b. Abutment temperature, measured in °C, is indicated by a green line 322b. Wheel temperature, measured in °C, is indicated by a blue line 323b. Abutment stress, measured in MPa, is indicated by the individual round data points 324b (for clarity, only one data point 324b is labelled).

Trial 4 was the first test using a W-25wt% Re abutment, which was expected to be less sensitive to wheel speed due to its superior hot working strength. Referring to Figures 29, 30, 31 and 32, the effective abutment stress levels observed during the experiments of Trial 4 were significantly higher than in the other trials (Trial 2, Trial 3.1 and Trial 3.2). In Trial 4, the rate of increase of abutment temperature was also much greater than before. The tests were terminated once the abutment temperature reached 700 °C as it was then clear that the abutment had failed. About 100 mm of 5 mm diameter rod was extruded but it had a very poor surface finish. It was discovered after Trial 4 that a change in tooling had resulted in the abutment being placed 500 µm closer to the wheel than in

previous trials. As the wheel groove still contained cold coated titanium from the previous trials, it is possible that the abutment failed before any powder was added to the machine. This would have changed the effective abutment profile resulting in erroneous readings for the abutment stress.

5 Figure 13 illustrates the wheel speed sensitivity of CP-Ti Grade 2 HDH powder during different phases of the process in Trial 2. Abutment stress, measured in MPa, is plotted against wheel speed, measured in RPM. The abutment temperature for each data point is indicated with reference to a temperature gradient bar 133 next to the graph. A dashed line 131 plotted on the graph represents a data fit for the start-up phase. A broken line 132 plotted on the graph represents a data fit for the
10 steady-state.

Figure 14 illustrates the wheel speed sensitivity of Ti-6Al-4V HDH powder fed in behind processed CP-Ti Grade 2 HDH powder in Trial 2. Abutment stress, measured in MPa, is plotted against wheel speed, measured in RPM. Diamond-shaped data points relate to the ramp-up phase.

15

Figures 15, 16A, 16B and 16C illustrate the wheel speed sensitivity of CP-Ti Grade 2 HDH powder during Trial 3.1. In each graph, abutment stress, measured in MPa, is plotted against wheel speed, measured in RPM. Diamond-shaped data points relate to the ramp-up phase.

20 Referring to Figure 15, the abutment temperature for each data point is indicated with reference to a temperature gradient bar 153 next to the graph. A dashed line 151 plotted on the graph represents a data fit for the start-up phase. A broken line 152 plotted on the graph represents a data fit for the steady-state.

25 Referring to Figure 16A, the abutment temperature for each data point is indicated with reference to a temperature gradient bar 163a next to the graph. A dashed line 161a plotted on the graph represents a data fit for the start-up phase. A broken line 162a plotted on the graph represents a data fit for the steady-state.

30 Referring to Figure 16B, the abutment temperature for each data point is indicated with reference to a temperature gradient bar 163b next to the graph. A dashed line 161b plotted on the graph represents a data fit for the start-up phase. A broken line 162b plotted on the graph represents a data fit for the steady-state.

35 Referring to Figure 16C, the abutment temperature for each data point is indicated with reference to a temperature gradient bar 163c next to the graph. A dashed line 161c plotted on the graph represents a

data fit for the start-up phase. A broken line 162c plotted on the graph represents a data fit for the steady-state.

The plots of wheel speed versus effective abutment stress in Figures 13-16C show how there is a counterintuitive drop in stress with increasing wheel speed as theorised above. There are two distinct regions, which demonstrate the difference between the effective abutment stress when grip or slip occurs between the wheel and feedstock. Referring to Figure 12, the start-up phase has significantly lower stress levels indicating that the wheel is slipping past the powder. As the process continues and the powder begins to consolidate the effective abutment stress is seen to increase for a constant wheel speed. The wheel starts to grip the densified material.

Powder Shear

With each wheel speed there is a maximum distance from the abutment face below which the shear ratio drops below unity. Above this distance the shear ratio is favorable for powder consolidation to occur within the flash gap. It is apparent that with increasing shear ratio there is an increase in stress within the powder bulk. Particles are then more likely to yield, deform, heat adiabatically resulting in particle-to-particle bonding. In the applicant's experimental set-up, this shear will be approximately 8.6% higher at the edge of the wheel than at the bottom of the groove.

Figure 17 is a graph of shear ratio plotted against distance below the abutment face for different wheel speeds. Shear ratio ($\text{mms}^{-1}/\text{mms}^{-1}$), on the y-axis, is a dimensionless ratio. The distance below the abutment face, on the x-axis, is measured in mm. A solid line 171 indicates a shear ratio of 1 (1:1 ratio). Data are plotted for five different wheel speeds, measured in RPM: 10 RPM indicated by line 172; 8 RPM indicated by line 173; 6 RPM indicated by line 174; 4 RPM indicated by line 175; and 2 RPM indicated by line 176.

The plot of shear ratio at certain points below the abutment face in Figure 17 shows two important regions. First is the intersection between 1:1 (line 171) and 2 RPM (line 176), from which it clear that the first 5 mm of drop has the potential to cause significant shear within the powder almost independently of wheel speed. When we compare this with Figure 7 it is clear that while abrasion of the abutment 71 by the powder has occurred down to approximately 5 mm, powder consolidation has not yet begun. The second important region is the intersection of 1:1 (line 171) and 4 RPM (line 175) at 19 mm. When compared with Figure 8, we see that when a wheel speed of 4 RPM is used consolidation can begin. The height of consolidated material down the die chamber face in Figure 8 is consistent with this intersection of 1:1 (line 171) and 4 RPM (line 175) in Figure 17. Data are only plotted up to a distance of about 30 mm from the abutment face in Figure 17 as there was a relief facet

in the abutment. Further consolidation of powder is impossible past this point due to an inability to feed in fresh powder to account for the rapid increase in flash gap size.

Microstructure

5 Optical-Polarised Light

The rods extruded from Trials 3.1, 3.2 and 4 were all less than 100mm in length and exhibited incomplete consolidation during the start-up phase of the Conform process.

10 Figure 18 shows a cross-section of rectangular rod taken from Trial 3.2 where the flow across the middle of the image can be seen. The flow of powder was much clearer than with samples of the 5mm round rod (not shown), possibly due to the lower processing temperatures allowing inadequate time for complete particle-particle bonding. Entrained particles of Stellite™ 21 are also visible on the right hand side of Figure 18, which particles having come from the fracture of the abutment.

15 Figure 19 shows a representative microstructure taken from a small rectangular area 181 towards the lower left corner of Figure 18. The average grain size is $3.2 \pm 0.4 \mu\text{m}$ in this region (Figure 19) but is considerably larger at $23 \pm 5 \mu\text{m}$ at the edges of the section (Figure 18). Without wishing to be bound by any theory, this distribution may be the result of the edges having experienced further shear at the die surface and spent a relatively long time in contact with the die while the sample was being
20 removed resulting in a greater propensity for recrystallisation than in the centre of the cross-section.

Figure 20 is a composite macrograph of a section taken through the die back-end discard from Figure 12 (right) indicating the bulk material flow just back from the die entrance. The macrograph was taken using polarized light. An arrow 201 indicates the extrusion direction. The die position 202 is
25 also indicated alongside Figure 20 to assist in interpretation. Material dead zones can be seen at the top and bottom of the image as areas of increased grain size where material has been forced to sit against the die chamber while fresh powder was extruded over the top of it.

Finite Element Modelling

30 Figure 21 is a simulated strain distribution profile within a Conform machine. The Conform machine comprises a grooved wheel 211, an abutment 212 configured to divert and deform workpiece material into a die chamber port 213 and a die 214 leading from the die chamber port 213. In Figure 21, strain is measured in mm mm^{-1} . The strain within the Conform machine is indicated with reference to a strain gradient bar 215.

35

Figure 22 is a simulated strain rate distribution profile within a Conform machine. The Conform machine comprises a grooved wheel 221, an abutment 222 configured to divert and deform workpiece

material into a die chamber port 223 and a die 224 leading from the die chamber port 223. In Figure 22, strain rate is measured in $\text{mm mm}^{-1} \text{s}^{-1}$. The strain rate within the Conform machine is indicated with reference to a strain rate gradient bar 225.

5 Figure 23 is a simulated stress field within a Conform machine. The Conform machine comprises a grooved wheel 231, an abutment 232 configured to divert and deform workpiece material into a die chamber port 233 and a die 234 leading from the die chamber port 233. In Figure 23, stress is measured in MPa. The stress within the Conform machine is indicated with reference to a stress gradient bar 235.

10

Figure 24 is a simulated temperature distribution within a Conform machine. The Conform machine comprises a grooved wheel 241, an abutment 242 configured to divert and deform workpiece material into a die chamber port 243 and a die 244 leading from the die chamber port 243. In Figure 24, temperature is measured in $^{\circ}\text{C}$. The temperature within the Conform machine is indicated with
15 reference to a temperature gradient bar 245.

Figure 25 is a simulated velocity distribution within a Conform machine. The Conform machine comprises a grooved wheel 251, an abutment 252 configured to divert and deform workpiece material into a die chamber port 253 and a die 254 leading from the die chamber port 233. In Figure 25,
20 velocity is measured in mm s^{-1} . The particle velocity within the Conform machine is indicated with reference to a velocity gradient bar 255.

Figure 26 shows simulated flow lines of tracked points through the abutment zone of a Conform machine. The Conform machine comprises a grooved wheel 261, an abutment 262 configured to
25 divert and deform workpiece material into a die chamber port 263 and a die 264 leading from the die chamber port 263. Four flow lines 265, 266, 267, 268 are shown in Figure 26. Two material dead-zones 269a, 269b can also be seen. In Figure 26, velocity is measured in mm s^{-1} .

Figures 21, 22, 23, 24, 25 and 26 show the variation in state variables in a half section of the
30 workpiece. The simulation was stopped once extrusion had begun and the temperature and stress fields had stabilised. The strain distribution (Figure 21) shows a large gradient from the wheel 211 groove on the right to the die 214 on the left. The strain magnitude is very high at between 10-15 mm/mm within the extruded rod.

35 Figure 21 shows the primary deformation zones within the Conform process. There is a primary zone that links the abutment 212 tip and top of the die chamber port 213 where the material undergoes intense shear, similar in position and magnitude to FEM simulations of equal channel angular pressing

(ECAP). There is a smaller secondary zone at the entrance to the die 214 where further strain can be induced within the edges of the rod.

The stress field (Figure 23) exhibits a peak at the wheel-workpiece interface where there is intense shear occurring. With a yield stress of approximately 345 MPa (AMS 4902 specification) all of the workpiece deforms plastically within the abutment zone. Within the die chamber port 233 to the left of Figure 23 the stress field is fairly constant before rising again around the die entrance.

The temperature distribution (Figure 24) shows that there is rapid heating of the workpiece within a few seconds of deformation beginning. The initial workpiece temperature of 20 °C rises to a peak of approximately 700 °C within the bottom of the abutment zone and over 900 °C within the flash gap. This may have implications for the real-life processing of titanium within the Conform process as it is possible that uncontrolled areas of the workpiece or flash will transform to beta phase at these high temperatures.

Figure 25 shows the velocity distribution within the abutment zone and helps to delineate the flow of the bulk material. There is an intense shear close to the wheel that is mirrored by the strain rate distribution (Figure 22) where the workpiece undergoes the majority of its deformation. The centre of the workpiece then moves downward under the pressure of fresh material above it before moving back up towards the die entrance. Both above and below the die are areas of slow moving material known as deadzones. Figure 26 shows two deadzones 269a, 269b, which were defined by velocity < 3 mm s⁻¹. The upper deadzone 269a demonstrated a circular flow where it might be possible for material that has spent a long time within the hot abutment zone to be fed slowly back into the extruded rod.

Discussion

The machine data from the experimental trials (Figures 29, 30, 31 and 32) show how small variations in different process parameters can greatly affect the successful extrusion of CP-Ti HDH powder. Wheel speed variations between Trial 2 and Trial 3.1 demonstrate that the wheel speed must be ramped up quickly to enable a drop in the measured torque. This is reflected in the drop in effective abutment stress and that Trial 2 produced a long length (16.7 m) of rod with consistent properties. While each of the Trials 3.1, 3.2 and 4 generally failed to produce a satisfactory extrusion, they nonetheless helped determine the limits beyond which it may not be possible to process CP-Ti HDH powder in a Conform machine.

The microstructure of the rod that was extruded is made up of fine equiaxed grains implying that significant recrystallisation has occurred. This is backed up by the simulated temperature profile in

Figure 24 where the temperature within the extruded rod reaches 700 °C. The grain sizes seen here are also consistent with those found in the 5 mm rod from Trial 2. The macrograph of the cross section of the rod in Figure 18 illustrates the unusual flow behaviour of the powder within the Conform process. It indicates that the powder particles undergo significant rearrangement and deformation before being extruded. It is possible that the strain evolved in the material generated from powder feed is significantly higher than that seen with rod feed in the finite element model.

Figure 27 is a graph of effective abutment stress calculated from torque measured on the wheel in truncated finite element simulations (as discussed above in relation to Figure 6). Abutment stress, measured in MPa, is plotted on the y-axis. Time, measured in seconds (s), is plotted on the x-axis. A solid line 271 shows data for a full simulation. Broken lines 272 (40 mm height), 273 (30 mm height), 274 (24 mm height), 275 (18 mm height), 276 (13 mm height), 277 (7 mm height) show data for the six truncated finite element simulations.

Figure 28A is a graph of average abutment stress over sample simulations plotted against wheel groove fill depth. Abutment stress is measured in MPa; fill depth is measured in mm. Truncated sample data 282 are indicated by cross-shaped data points with error bars towards the lower left of the graph. The data point 281 towards the upper right of the graph represents data from the full simulation.

Figure 28B is a graph of average abutment stress over sample simulations plotted against grip length for four different metallic powders: Al 1100; Ti 6-4; CP-Ti; and Ti-5553. Abutment stress is measured in MPa; grip length is measured in mm. A red line 283 fits the data for Al 1100 powder. A blue line 284 fits the data for Ti 6-4 powder. A green line 285 fits the data for CP-Ti powder. A purple line 286 fits the data for Ti-5553 powder.

The variation of effective abutment stress with fill depth in Figure 28A exhibits a linear relationship. As the grip length increases up to a maximum of 150 mm due to the tooling geometry the abutment stress increases steadily (as shown in Figure 28B). For each of these simulations the stress level was fairly constant, only changing significantly around remeshing steps. Measurement of the grip length within the full simulation also allowed comparison of the effective abutment stress with the truncated samples as represented by the far right hand point 281 on Figure 28A. It is found that the abutment stress can be estimated to be $\sigma_{abut} = 10.4h$ where h is the height of fully consolidated material above the abutment and in full contact with the wheel. In practice, this is likely to be a lower limit to this height as there will be an extra contribution to the wheel torque from partially consolidated powder above the layer that is fully dense.

During the addition of fresh cold powder a number of things can occur within the groove. The new cold powder flows freely according to its initial morphology and has a nominal contact area with the wheel and the entry blocks. This initial contact area results in certain level of resistance to extrusion. As the powder heats up through contact with the tooling, particle-particle contact and particle deformation the friction conditions within the groove change. The net effect is to considerably increase the overall bulk powder temperature and its contact area with the tooling. During this secondary phase the primary shear occurs higher up the groove in a larger section of loose powder. At first this will consolidate powder close to the abutment and begin to build the pressure required for extrusion through the die. As the powder densifies firstly during particle rearrangement and then particle deformation there is an increase in effective pressure normal to the surfaces of the wheel and shoe. This causes an increase in interface friction in line with Coulombs Law. The height above the abutment which is fully consolidated increases as the process continues resulting in an increase in grip length between the powder and wheel. This continues to increase until enough of a grip length causes the extrusion pressure to be reached.

For a given grip length between the wheel groove and the powder there is an increase in extrusion pressure as this contact area increases. With powders of copper and aluminium the tooling is reasonably tolerant of the process startup conditions. Evidence from process data files of low strength alloys ($\sigma_y < 200\text{MPa}$) indicate that the initial wheel speed is not important in obtaining a successful extrusion product. However, when processing high strength alloys such as commercially pure titanium the processing window is far smaller. The tooling materials, in particular the abutment have a tendency to yield early in the process when using titanium powder feedstock. This occurs frequently when using a relatively slow wheel speed of 2-4 RPM but not when the wheel speed is driven above 5 RPM. Analysis of the process data files indicates that the stress normal to the face of the abutment can be an order of magnitude greater at 2 RPM than 6 RPM. For a wheel speed of 2 RPM powder particles would be deformed at a much lower strain rate and have a greater chance to lose more heat evolved from mechanical work. Friction conditions would also be more stable due to the relative lack of particle rearrangement when compared with higher wheel speeds.

The grip between the wheel and the powder at lower wheel speeds needs be strong enough to resist slippage. If this is the case then lower wheel speeds would result in longer effective grip lengths between the wheel and the powder. As the wheel speed increases for a given powder bulk formation there is a maximum wheel speed to enable a successful grip between the wheel and feedstock. Below this speed, the maximum grip length is determined by the powder feed rate and the tooling geometry. If the wheel groove is only enclosed up to a certain height, powder fed on top of this would spill out of the sides of the groove and the grip length wouldn't increase. If the wheel speed increases above

this maximum, then appreciable slippage will occur between wheel and feedstock reducing the effective torque required by the wheel to maintain a constant angular velocity.

With rod feedstock wheel slippage is detrimental to extrusion performance as high friction is required to develop the main extrusion pressure. If slippage occurs with powder feedstock there is an immediate shear at the powder-wheel interface resulting in a high degree of particle rearrangement, frictional heating, deformation and adiabatic heating. This can heat up the powder while maintaining a minimal normal stress on the abutment face. As this slippage continues the powder will heat up within the process and the particles flow stress will drop considerably. The particles will then preferentially deform rather than rearrange resulting in the aforementioned increase in groove-feedstock contact area and an increase in grip length. This balance of contact area, grip length, wheel speed and feedstock material flow stress all works together to minimise the effective stress on the abutment when extrusion radial to the wheel occurs.

The results from the truncated FEM simulations can be applied to the data gained from the experimental trials to determine what the expected powder fill depth is within the groove. This is something that is impossible to determine experimentally for a typical Conform machine, due to the enclosing shoe obscuring the view of the abutment zone. This data allows the estimation of the required powder feed rate for a certain wheel speed. This is an important finding as it is critical with wheel speed sensitive powders such as titanium that the wheel groove is not allowed to fill up to its maximum to prevent failure of the abutment. It is also equally important to ensure enough powder is fed in at a rate appropriate for the current wheel speed so that the extrusion pressure is maintained and that material lost in the flash and extrudate is compensated for. If the powder feed rate is too slow the abutment zone will lose more material than is being added until extrusion stops. This would be detrimental to the rod properties as it sits in the heated die and forms stick/slip lines along its surface. Such lines were visible on the rod from Trial 2, which indicated that the powder feed may have been too low for a consistent extrusion but not high enough to cause the abutment to yield.

Two stages of bulk powder deformation can be seen to occur within the process graph shown in Figure 13 where the slip curve is at the bottom of the graph. Here the wheel grip with the powder is inadequate to produce a significant amount of pressure. As the wheel speed increases there is a slight drop in the effective abutment stress but it remains fairly independent of wheel speed at 100-150 MPa, which is consistent with a velocity independent friction model. The upper curve in Figure 13 represents the stress levels during active extrusion. If we use the results determined from the truncated finite element (FE) simulations it can be shown that the change of effective abutment stress with constant wheel speed on this upper curve implies a change in powder feed rate. When we apply this theory to Trial 3.1 in Figure 15 it can be seen that the powder feed rate during the start-up of this trial

was much higher than in Trial 2 resulting in an increase in effective grip length and hence abutment stress of an order of magnitude. This correlates with the failure of the abutment early within this trial when running the wheel at 2-4 RPM. The failure of the abutment during Trial 3.2 only occurred after a short length of rod was extruded.

5

Figure 16 shows much lower effective abutment stress values at all wheel speeds possibly indicating that the powder feed rate was slower during this trial. The drop in abutment stress with increasing wheel speed could also be attributed to the effect of having a constant powder feed rate. With a steady feed rate the powder would fill the groove much higher with lower wheel speeds resulting in a greater stress on the abutment.

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Conclusions

It was originally thought from observations of the process graphs (Figures 13, 14, 15 and 16) that there may be a minimum wheel speed required for successful extrusion of CP-Ti HDH powder within the Conform process. Without wishing to be bound by any theory, it may be assumed that a faster wheel speed would induce greater heat generation within the powder, lowering the flow stresses and improving particle-particle bonding.

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Additionally or alternatively, our results indicate that for the successful continuous rotary extrusion (e.g. using the Conform process) of CP-Ti HDH and possibly other high strength powders it may be critical to not exceed a maximum fill depth within the wheel groove. It is possible to maintain the fill depth below this critical value by tailoring the powder feed rate depending on the wheel speed.

20

The critical fill depth was found to be approximately 30-35 mm based on evidence from the trials data graphs where the abutments failed and the truncated FE simulations. It also corresponds to the boundary observed in the simulations where samples of 30 mm and below would fail to deform significantly. This critical fill depth is not unique to Conforming of CP-Ti HDH.

25

There will be a critical fill depth for all other metallic particulate feedstocks. Without wishing to be bound by any theory, it may be postulated that the critical fill depth (and hence the predetermined level, which will be no more than the critical fill depth) may be determined to a significant extent by the powder morphology, alloy chemistry and yield strength of the material. The critical fill depth will also vary depending on the grip length (affected by groove size), die size and die chamber port size. For example, with CP-Ti HDH powder (of size fraction 45µm-150µm) the critical fill level has been found to be 30 - 35 mm with a 17.5 mm high and 10 mm deep arched die chamber port, 12 mm wide groove and 5 mm round die.

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However, it appears that the critical fill depths for alloys of aluminium and copper are significantly higher than that allowed by the typical geometry of a Conform machine; hence prior experience of processing aluminium and copper using the Conform process has not provided persons skilled in the art with any appreciation of the importance of fill depth.

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By applying this theory, it may be possible to successfully Conform CP-Ti HDH at lower wheel speeds than was previously thought by using a lower powder feed rate to ensure the critical fill depth is not exceeded. Processing at lower speeds may have the added benefit of lower processing temperatures and die wear, hence allowing for longer tool lifetimes. There will be a lower limit to this, as the required extrusion pressure would increase with decreasing powder temperature at lower wheel speeds.

Modifications to the current typical Conform tooling geometry may also make it possible to process CP-Ti HDH powder without the need for a metered powder feed. For instance, a relief facet added to the die chamber port at the height of the critical fill depth could ensure that the critical fill depth was never exceeded as excess powder would be ejected from the sides of the groove.

It is noted that the data used to predict the grip length with CP-Ti HDH powder is based on simulations using a single wheel speed and doesn't allow for changes in material flow stress. The workpieces within the truncated simulations did not heat up or deform appreciably resulting in a fairly constant resistance to deformation throughout the entire simulation. In reality the powder heats up rapidly and its flow stress will drop equally as quickly.

Further simulations to determine what effect the change in flow stress may have on the abutment stress - fill depth relationship may be required to enable successful extrusion after the initial start-up phase of the Conform process. Simulations using workpiece materials with different flow stresses may also help to predict whether there is an upper limit in terms of the strength of the powder that is possible to be processed using the current Conform 315i machine. The upper limit will be reached when the minimum grip length required to generate enough extrusion pressure exceeds the maximum fill depth before the stress on the abutment causes its failure. With a critical height of 30-35 mm for CP-Ti HDH, there is only a surplus of 17 mm in fill depth before the powder falls below the die chamber port. This potential overlap of zones could be mitigated by running the wheel at faster speeds to generate heat and lower the flow stresses within the powder. The only requirement would be a very low powder feed during the startup phase. The person skilled in the art would be able to carry out any necessary further work as a matter of trial and error. With the benefit of the disclosure of this patent application, without exercising any inventive skill he would be able to produce a titanium wire or rod from a particulate feedstock by a continuous, solid-state extrusion process.

On a macrostructural and/or microstructural level, the grain structure and texture generated from continuous rotary extrusion (e.g. Conform) may be unique for titanium powder. No other known process can produce rod with this macrostructure, microstructure and texture from titanium in any form.

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Referring to Figure 33, the microstructure has a bimodal distribution of fine-grained regions (e.g. as indicated at 331a and 331b) and coarse-grained regions (e.g. as indicated at 332a and 332b) with respect to the flow pattern. The exact grain sizes in the distribution will be determined by the extrusion temperature and rate of cooling post-extrusion.

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Fine grains may tend to form in a central line and in arced regions through the rod cross section. Typically, coarse grains may form in a ring around the circumference of the rod and in-between the fine grained regions.

15 Figure 34 shows cross sections of rod that was continuously rotary extruded from pure aluminium powder. The images in Figure 34 illustrate the range of flow patterns observed in softer materials. In contrast, a rod continuously rotary extruded from titanium typically may have a stronger central flow region with a smaller circular “onion skin” around the outside.

20 Figure 35 includes two micrographs taken from a section of a CP-Ti rod manufactured by the Conform process from a CP-Ti powder. The left-hand micrograph is of a coarse-grained region of the section. The right-hand micrograph is of a fine-grained region of the section. In the example shown in Figure 35, the coarse grains typically measure more than 20 μm across while the fine grains typically measure less than 5 μm across. However, as noted above, while a bi-modal distribution of
25 coarse-grained regions and fine-grained regions may be characteristic for titanium rod or wire manufactured in accordance with the invention, the size of the coarse grains and the fine grains will vary depending, for example, on the extrusion temperature and post-extrusion cooling rate.

Figure 36 shows the macrostructure of a Conformed CP-Ti rod manufactured in accordance with the
30 invention, which has subsequently undergone a two-hour heat treatment. The characteristic bi-modal distribution is still visible. Due to titanium’s recrystallisation behaviour the unique macrostructure remains even after heat treatments.

The applicant’s experiments have so far yielded many important results that were to make the
35 Conform of CP-Ti HDH powder reproducible.

It is envisaged that lengths of rod, bar or wire in excess of 20 m may be produced from titanium particulate feedstock, e.g. CP-Ti HDH powder, in accordance with the invention.

Other metallic particulate feedstocks with different chemistries including Ti-6Al-4V, Beta III and Ti-30%wt Ta, mixed CP-Ti and Ti-6Al-4V machine turnings may also be processed in accordance with the invention.

Accordingly, it will be appreciated that the invention may be applicable to the manufacture of articles from metallic particulate feedstock comprising higher strength alloys (compared with aluminium and copper alloys), including titanium, tantalum and zirconium alloys.

Typically, wheel velocity and feed rate may be coupled inputs. For example, if the feed rate increases, then the wheel velocity may need to increase to expel more extrudate/flash and reduce the fill height back to the critical value. This increase in wheel speed would also increase the temperature of the processed feedstock, which would be mirrored with slight increase in measured tool temperature. The shorter the residence time of the powder moving from the groove to the die and/or a quicker post-extrusion quench time may also affect the amount of post-extrusion annealing the rod will undergo when it emerges from the die at extended temperature.

Advantageously, an overflow facet may provide a safety measure so that the critical fill height isn't exceeded. The provision of an overflow facet may also provide a visual indication to the machine operator that the critical fill height had been exceeded.

Advantageously, any excess powder captured in the overflow facet could be re-sieved (graded) and used again depending on the microstructural requirements of the final extruded product.

Measuring the motor current and/or torque may provide a way of monitoring the stress within the groove and die chamber. The critical value of the motor current and/or torque will vary depending on feedstock morphology, flow stress, feedstock temperatures throughout the process, tool temperatures and tooling geometry. It may then be possible to infer how changes in these factors will affect the stress on the abutment, fill within the wheel groove and hence the change in motor current.

Articles manufactured in accordance with the invention may undergo further processing steps, in order to produce a product. Such further processing steps may include mechanical, thermal and/or chemical processes, e.g. cold drawing, coiling, shot peening, annealing or pickling. The product may comprise a spring or a wire. The spring may be an automobile engine valve spring or a spring for a vehicle suspension system.

In an embodiment, CP-Ti wire may be produced in accordance with this invention by solid-state continuous extrusion from a particulate feedstock and subsequent wire drawing to produce wire with a preferred diameter. For instance, 3.5 mm diameter titanium wire may be suitable for coiling into automobile engine valve springs.

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Titanium valve springs are more efficient than steel and their use would contribute to vehicle CO₂ emission reduction.

The use of titanium suspension springs may also provide beneficial vehicle weight and emissions savings.

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Another example product that could be manufactured in accordance with the invention is landing gear springs for aircraft.

Other examples of potential products may include zirconium fuel cladding for the nuclear industry and feedstock for welding wire.

15

Still further examples of potential products may include: bolts rivets or other fixing structures; wire products such as welding wire, dental wire, spring wire forms; engine valves; connecting rods; or parts of a structure such as a building or a vehicle, e.g. an automobile (such as a car, a lorry or a bus) or an aircraft.

20

For spring applications, beta-type or beta-rich alloys typically may be preferred due to their low stiffness high strength properties. Such alloys are termed beta alloys as they are predominantly beta (body centre cubic crystal structure) phase due to the alloy addition of beta stabilisers such as V, Mo, Nb, Fe, Ta that stabilise the high temperature beta allotropic phase at room temperature. Beta alloys are strengthened by the precipitation of a fine scale (nano-structured) secondary alpha phase. The texture developed in the beta phase (and allotropic transformation relationship with the secondary alpha during a subsequent ageing treatment) will be determined by the severe deformation which the material undergoes in the manufacturing process of the invention.

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For general structures, alpha + beta type alloys (such as the most common alloy Ti-6Al-4V) may be preferred due to a combination of high stiffness and high strength. These alloys have more alpha phase stabiliser such as Al, O and typically around 90% of the structure is alpha at room temperature, with the remainder being stabilised beta phase. Similar to CP-Ti, strong alpha phase textures will be created during the manufacturing process of the invention.

35

Furthermore, embodiments of the invention may comprise an improved continuous extrusion apparatus and/or improved control systems and/or methodologies for continuous extrusion processes. Such an improved control system or methodology may be applied to an existing continuous extrusion apparatus. Similarly, modifications may be made to an existing continuous extrusion apparatus in order to improve its performance in accordance with the current invention.

A machine for carrying out the method according to the invention may comprise a sensor and feedback loop. The provision of a sensor and feedback loop may be advantageous, since, in the case of a Conform machine, an operator typically couldn't be expected to manually control the powder flow rate and wheel speed. The feedback loop may be based on measurements of the motor current and/or wheel torque (if the continuous extrusion machine has a wheel) and, advantageously, may be for retro-fitted to an existing continuous extrusion machine, e.g. a continuous rotary extrusion machine.

In an embodiment, a continuous extrusion machine according to the invention may be fitted with one or more pressure sensors, in order to directly measure the fill height and progress of the initial consolidation process. For instance, in the case of a Conform machine, the one or more pressure sensors may be fitted on to the stationary shoe.

A machine for carrying out the method according to the invention may, in some embodiments, be provided with a hopper fitted with a meter or other measuring device operable to measure the flow, in use, of metallic particulate feedstock into the groove. By measuring the powder flow rate into the groove for a given powder and machine, it may be possible to calibrate a mass flow rate that could be generalized to other powders and machine designs.

It will be appreciated that the invention may provide a method for continuous production of high strength, e.g. titanium, rod or wire from particulate metallic feedstocks. Furthermore, the invention may provide articles and products manufactured in accordance with such a method.

In particular, a processing window has been developed to allow the production of titanium or titanium alloy rod or wire via a continuous solid-state rotary extrusion method. The understanding that underpins the development of this processing window may also apply to the production of other high strength rod or wire via a continuous solid-state rotary extrusion method.

The applicant's knowledge of the process window may enable continuous extrusion of metallic, e.g. titanium, particulate feedstock to wire or rod using any continuous extrusion machine.

Advantageously, the process window consistently allows successful production of metallic, e.g. titanium, wire or rod from particulate feedstock, whilst maintaining tool/die integrity. In contrast, previous standard knowledge and understanding of continuous extrusion has been insufficient to enable titanium particulate to wire production as the standard rod feedstock operates outside of the particulate process window.

Furthermore, the invention could be used to modify existing continuous extrusion processes (e.g. a continuous rotary extrusion process such as Conform), e.g. of particulate feedstocks comprising aluminium or copper or alloys thereof such as brass. Thus, for example, it may be possible to modify and/or control the adiabatic heating and/or pressures in the feedstock and hence the microstructural evolution of the extruded product.

Changing the groove geometry (e.g. cross-section) typically will change the required and/or critical fill depth, since the grip surface area per unit length of the groove will vary. Furthermore, for a given continuous extrusion machine and metallic particulate feedstock, typically there will be a relationship between the groove shape/size, die shape/size, die chamber shape/size, flash gap, powder composition, powder tap density, powder morphology and the fill height. Changes to the groove geometry may occur as a result of changing the groove itself or from changing tooling such as shoe/entry plates situated above the die chamber that may feature protrusions into the groove.

In an embodiment, the extrusion pressure in the die may be of the order of at least 400 MPa and/or up to 600 MPa for CP-Ti HDH powder. However, the extrusion pressure may vary significantly depending on the stage of the process. For instance, the extrusion pressure may be much higher during the process start up than during steady state processing. For instance the local pressure within the groove that generates the extrusion force is higher than that within the die chamber port as the material is severely deformed and at a lower temperature. At the die exit the local pressure is still high but lower than the groove due to the much greater temperatures. Ideal simulations with an unrestricted upset fill height have shown the pressure within the die chamber port to be ~550MPa and in the groove to be ~950-1000MPa. This reduces to a groove pressure of ~550-600MPa when the fill height is constrained at 35mm.

The particulate feedstock may have a wide range of particle sizes and morphologies. For a given metallic particulate feedstock, if it is too fine, then the powder may not flow into the machine as van der Waals forces and air pressure may become a defining factor in the flow rate. A very fine powder may also have a tendency to spark and ignite, depending on its chemistry. Conversely, if the powder is too large, then the powder particles may tumble in the groove without interlocking, which is a

necessity for particle-particle diffusion bonding. Similarly, if the particles are too spherical, then particle interlocking may not occur on a large enough scale to begin consolidation.

- In an embodiment, feedstock particles may be selected according to the size of flash gap between the abutment and the groove. In the method according to the invention, there needs to be a certain level of interlocking but only after the particle feedstock has been warmed by the tools. If the particles create a bridge across the flash gap too soon, then there could be too much local pressure on the abutment tip, potentially causing premature tool failure.
- 10 Many different sizes and shapes of die may be used with the invention, in order to produce article having a range of profiles and sizes. For instance, round dies having a diameter of up to or at least 5 mm or up to or at least 10 mm may be used. Rectangular dies having a range of profiles may be used. For instance, rectangular dies having dimensions of at least and/or up to 3 mm and/or at least and/or up to 9 mm may be used. Alternatively, larger round or rectangular profiles may be used.
- 15 Alternatively or additionally, dies having more complex profiles, e.g. L, T, U, I, X, W and tube profiles, may be used.

Various further modifications to the example embodiments described herein will be apparent to the person skilled in the art without departing from the scope of the invention.

Claims

1. A method of manufacture of an article by continuous solid-state extrusion, the method comprising:
 - 5 providing a metallic particulate feedstock material;
 - feeding the particulate feedstock material at a feed rate into a groove moving with a velocity;
 - transporting the material in the groove to a diverter configured to divert at least a portion of the material into a die chamber port leading to at least one extrusion die;
 - 10 and
 - extruding material through the extrusion die thereby producing the article;wherein the level of particulate material in the groove is controlled such that sufficient extrusion pressure is maintained without being such as to cause a failure of the diverter.
- 15 2. A method according to claim 1, wherein the level of particulate material in the groove is controlled such that it does not exceed a predetermined level, the predetermined level being selected so as to prevent the diverter from failing.
3. A method according to claim 1 or claim 2, wherein controlling the level of particulate
20 material in the groove comprises controlling the feed rate and/or the velocity of the groove.
4. A method according to claim 1, claim 2 or claim 3, wherein a relief facet associated with the groove provides an overflow for excess particulate material, thereby preventing, in use, the level of particulate material in the groove from exceeding a critical fill height.
- 25 5. A method according to any one of claims 1 to 4, wherein the extrusion die is oriented at an angle to the groove.
6. A method according to claim 5, wherein the extrusion die is oriented at an angle of 30 degrees
30 or more to the groove, e.g. the extrusion die is substantially perpendicular to the groove.
7. A method according to any one of the preceding claims, wherein the groove has a curved base, e.g. the groove is semi-circular in cross-section.
- 35 8. A method according to any one of the preceding claims comprising measuring a current to a motor causing the groove to move, and, optionally, adjusting the feed rate and/or the velocity of the groove in response to the current measurement.

9. A method according to any one of the preceding claims, wherein the groove is provided on a wheel.
10. A method according to claim 9, wherein the wheel is rotated at a speed of 5 RPM or more,
5 e.g. 6 RPM or more.
11. A method according to any one of the preceding claims, wherein the particulate feedstock material comprises titanium, tantalum, zirconium, niobium, aluminium or copper.
- 10 12. A method according to any one of the preceding claims, wherein the particulate feedstock material has an average particle size of up to 2 mm.
13. A method according to any one of the preceding claims, wherein the article comprises a rod or a wire or strip.
- 15 14. A method according to any one of the preceding claims, wherein the particulate feedstock feedstock material is fed into the groove at a temperature of no more than 100°C.
- 20 15. A method according to any one of the preceding claims, wherein the particulate feedstock material is fed into the groove at ambient temperature.
- 25 16. A method according to any one of the preceding claims, wherein the method is carried out in air.
17. A method of manufacture of a product comprising: providing an article manufactured according to any one of claims 1 to 16; and subsequently processing the article to form the product.
- 30 18. A method according to claim 17, wherein the product comprises: a spring; a bolt, a rivet or other fixing structure; a wire product such as welding wire, dental wire, spring wire forms; an engine valve; a connecting rod; or part of a structure such as a building or a vehicle, e.g. an automobile (such as a car, a lorry or a bus) or an aircraft.
- 35 19. An apparatus for manufacture of an article by continuous solid-state extrusion of a particulate feedstock material comprising a movable groove, the movable groove being operable to transfer material to a diverter configured to divert at least a portion of the material into a die chamber port leading to at least one extrusion die, wherein the apparatus further comprises a

relief facet configured to receive excess material from the groove to ensure that the level of material within the groove does not exceed a predetermined amount.

20. An apparatus according to claim 19, wherein the groove is provided on a wheel.

21. An apparatus according to claim 19 or claim 20, wherein the groove is bounded by a stationary shoe along at least a portion of its path to the diverter.

22. A method of controlling a solid-state continuous extrusion apparatus, the apparatus comprising a movable groove for transporting, in use, particulate feed material, to a diverter and an electric motor operable to cause the groove to move, the method comprising measuring a current to the motor, and comparing the measured current with a reference value.

23. A method according to claim 22 comprising the step of varying, in response to measuring the current to the motor, the speed of movement of the groove and/or the rate of feeding the particulate feed material into the groove.

24. A method according to claim 22 or claim 23 comprising comparing the measured current with a reference value.

25. A method of controlling a solid-state continuous extrusion apparatus, the apparatus comprising a rotatable wheel with a groove for transporting, in use, particulate feed material, to a diverter and a motor operable to cause the wheel to rotate, the method comprising measuring a torque on the wheel.

26. A method according to claim 25 comprising the step of varying, in response to measuring the torque on the wheel, the speed of rotation of the wheel and/or the rate of feeding the particulate feed material into the groove.

27. A method according to claim 25 or claim 26 comprising comparing the measured torque with a reference value.

28. A method according to claim 25, claim 26 or claim 27 comprising measuring the current to the motor.

29. A continuously extruded wire, rod or strip, typically comprising titanium or a titanium alloy, wherein the wire, rod or strip has, in cross-section, a microstructure comprising a bi-modal

distribution of fine-grained regions and coarse-grained regions with respect to the flow pattern.

- 5 30. A product or component comprising a continuously extruded wire, rod or strip, typically comprising titanium or a titanium alloy, wherein the wire, rod or strip has, in cross-section, a microstructure comprising a bi-modal distribution of fine-grained regions and coarse-grained regions with respect to the flow pattern.

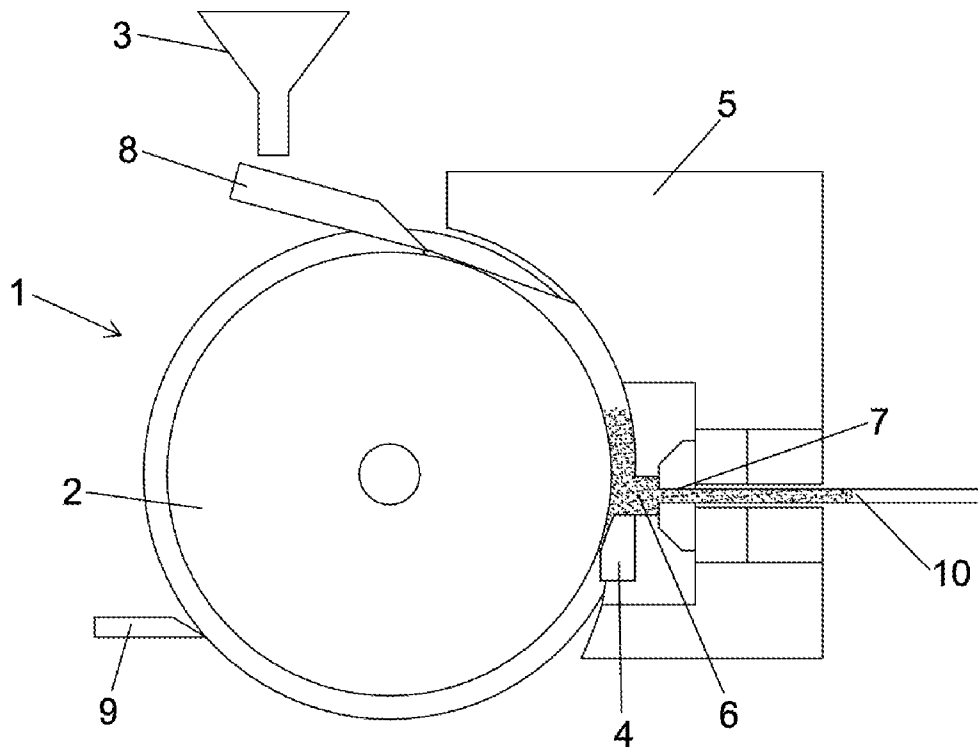


Figure 1

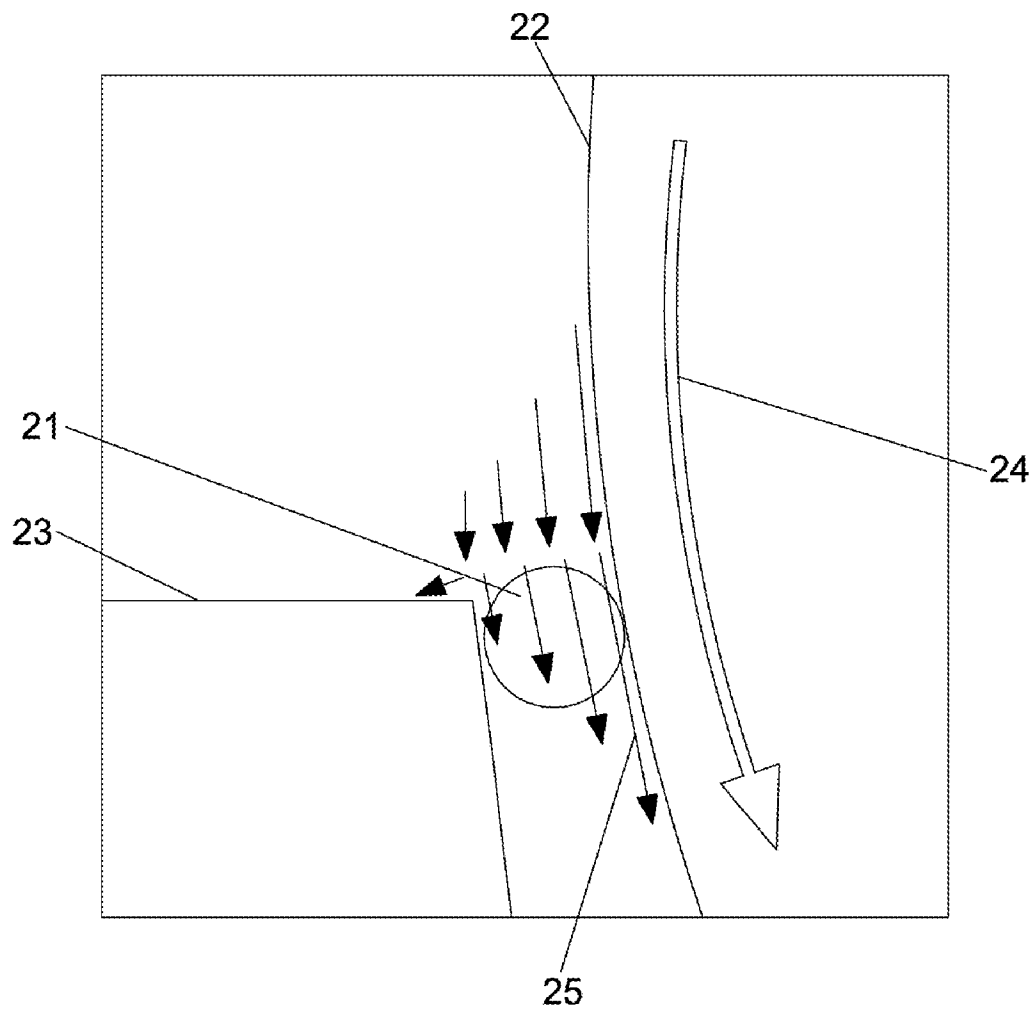


Figure 2

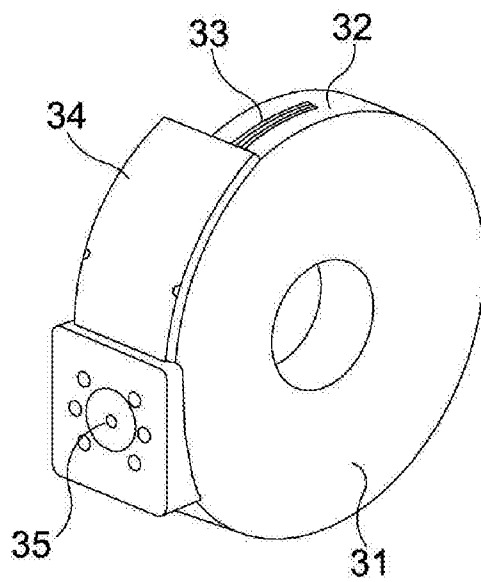


Figure 3

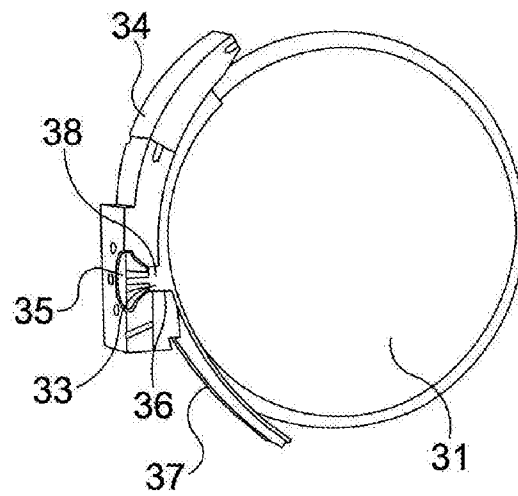


Figure 4

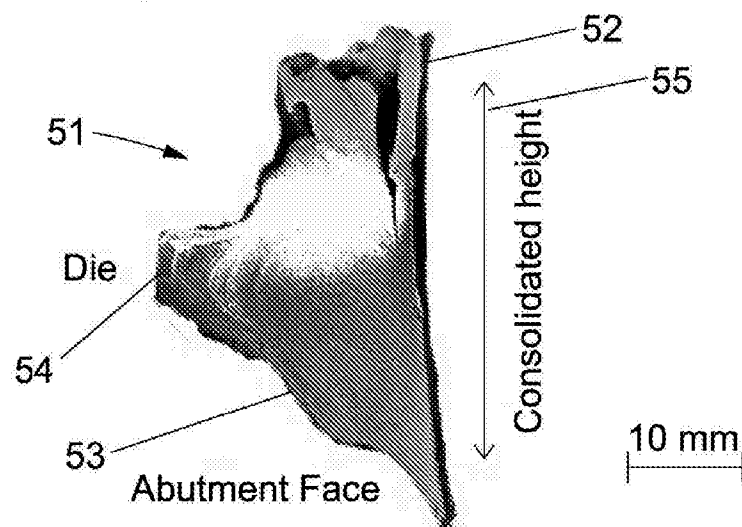


Figure 5

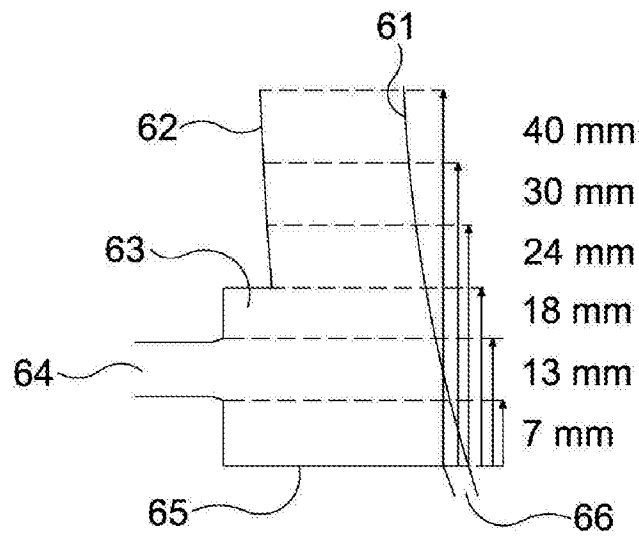


Figure 6

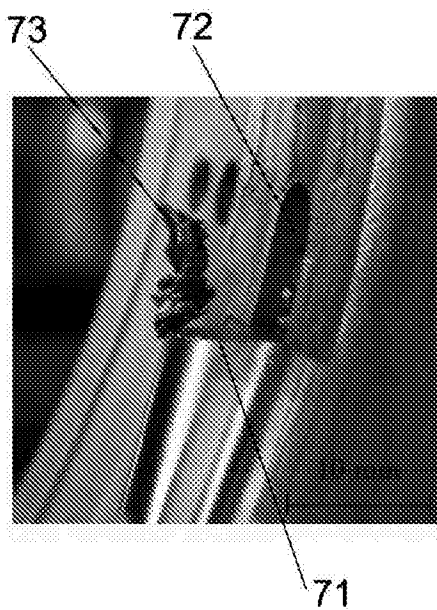


Figure 7

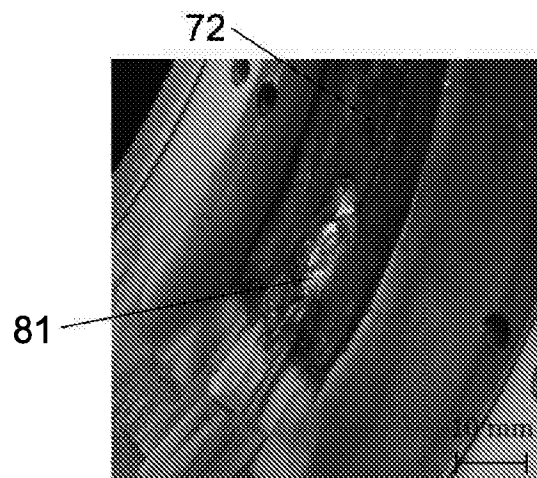


Figure 8

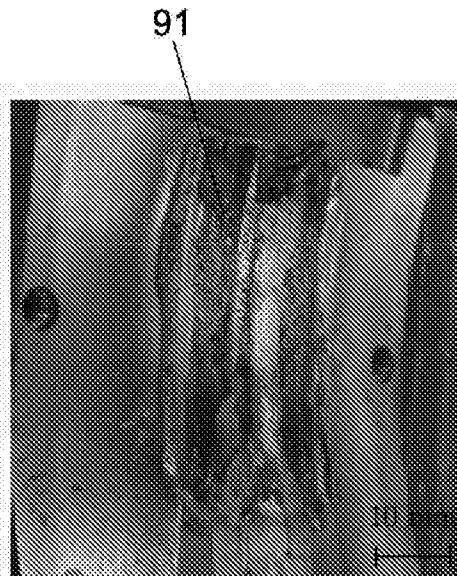


Figure 9

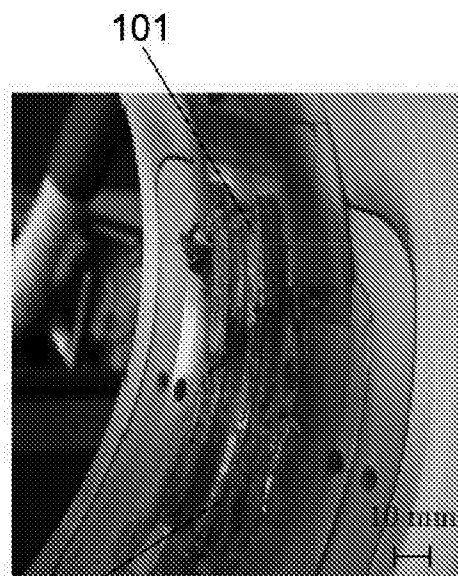


Figure 10

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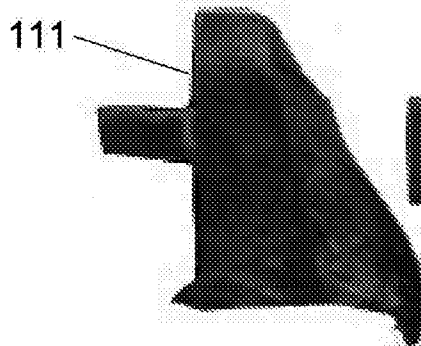


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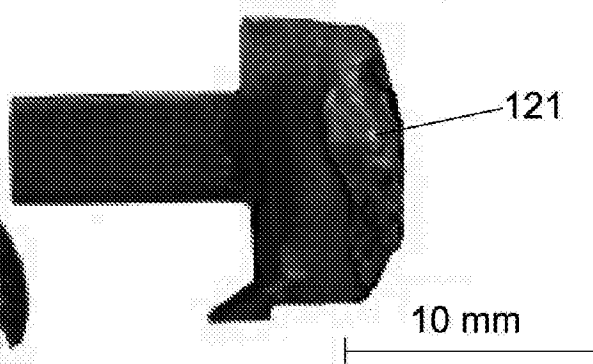


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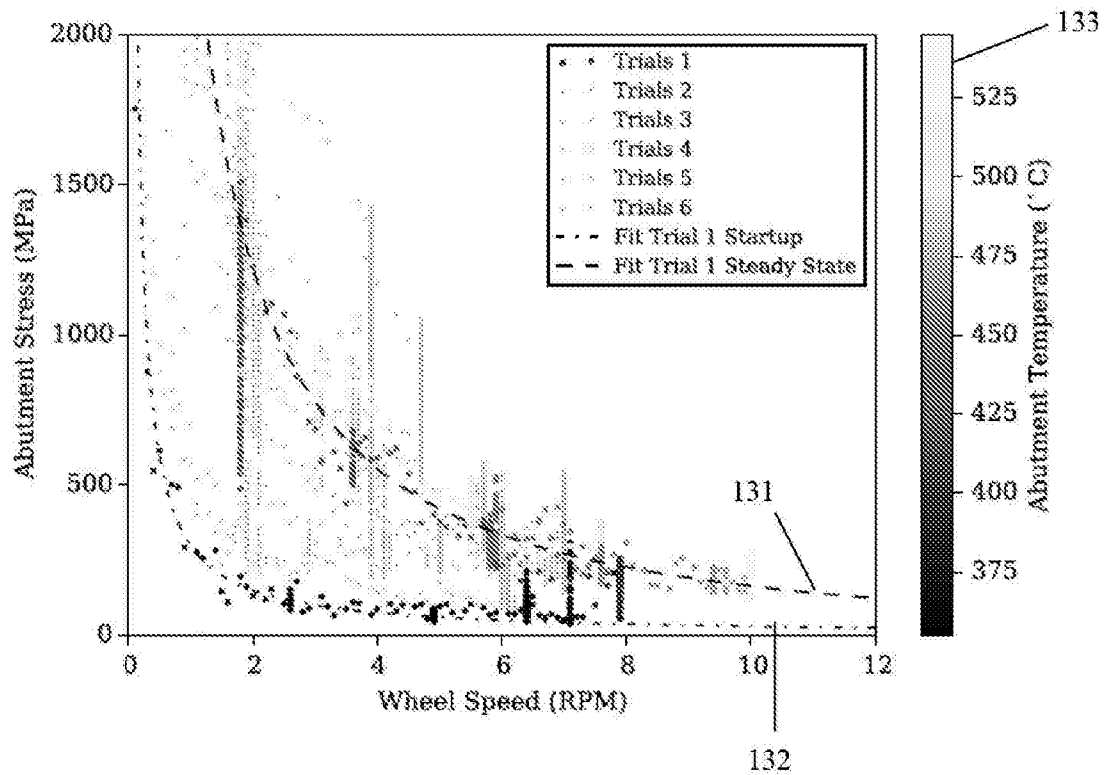


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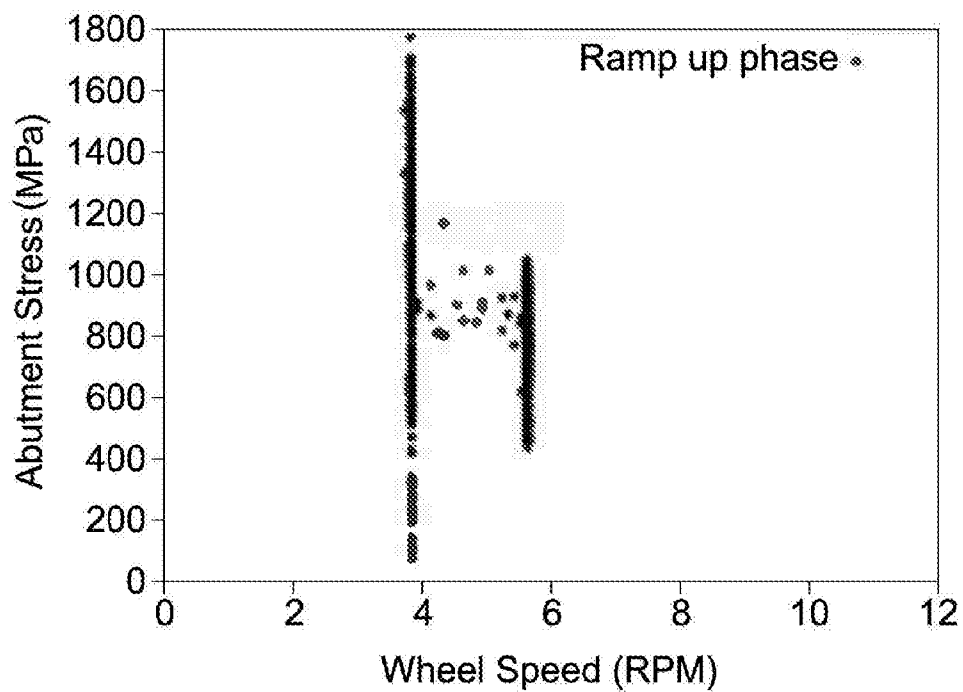


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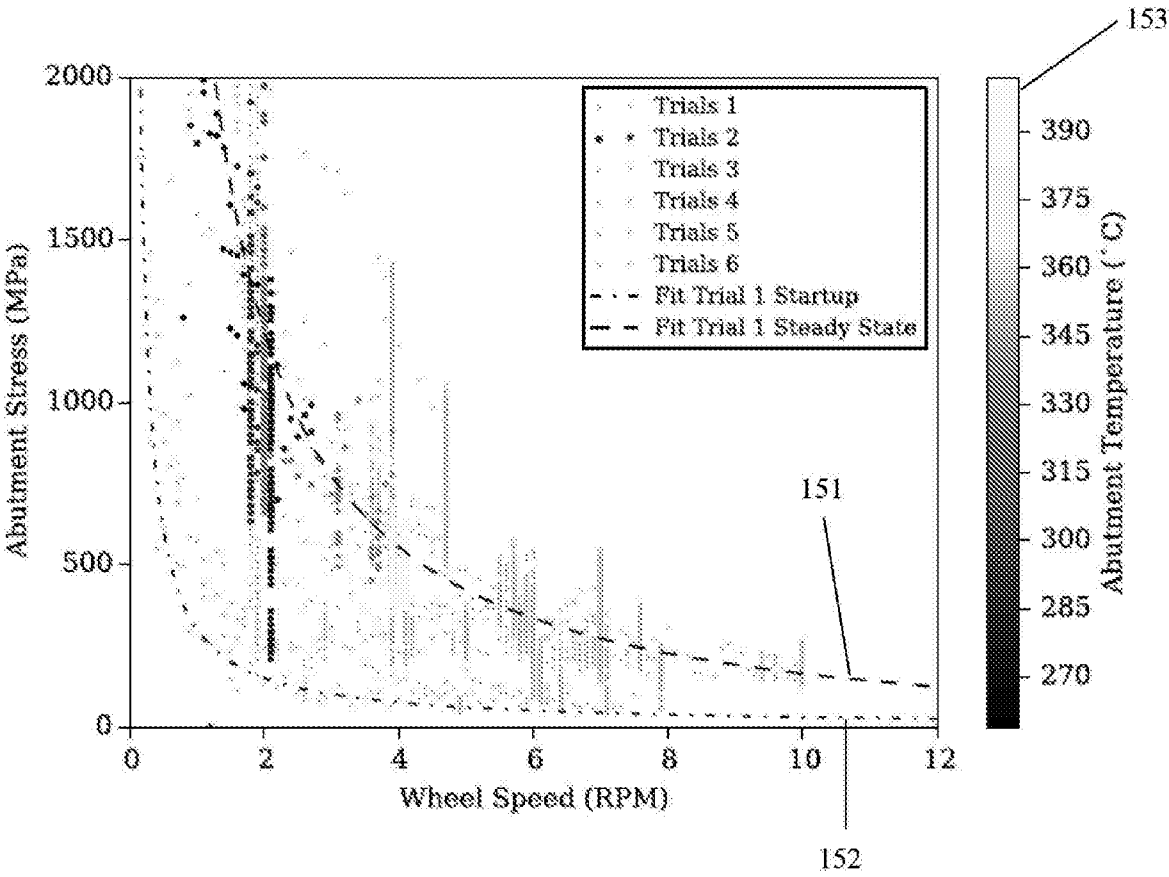


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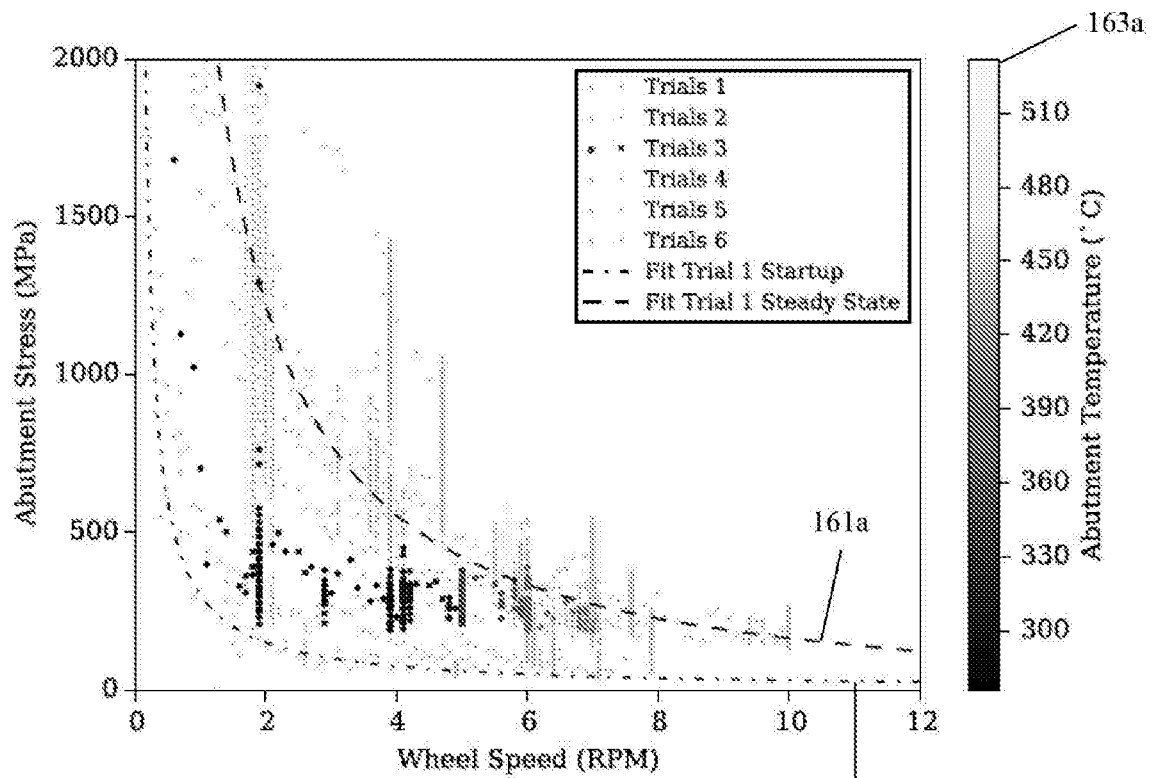


Figure 16A

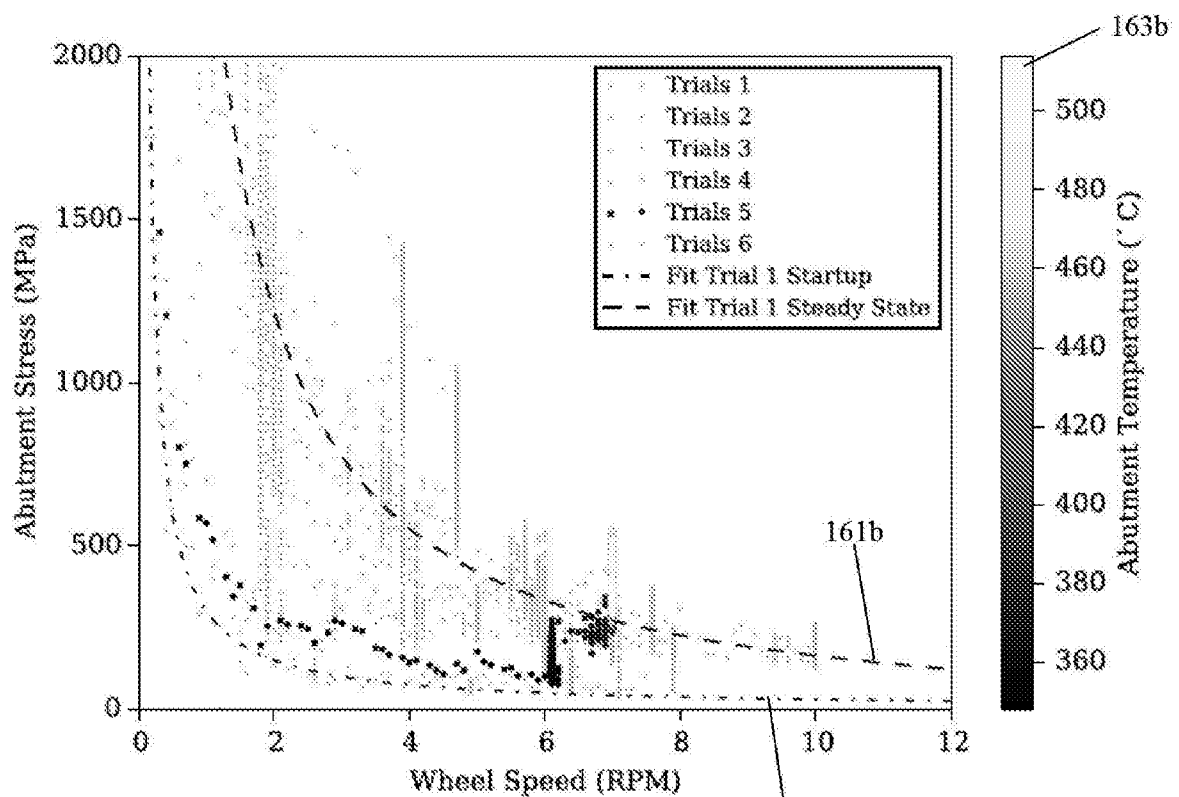


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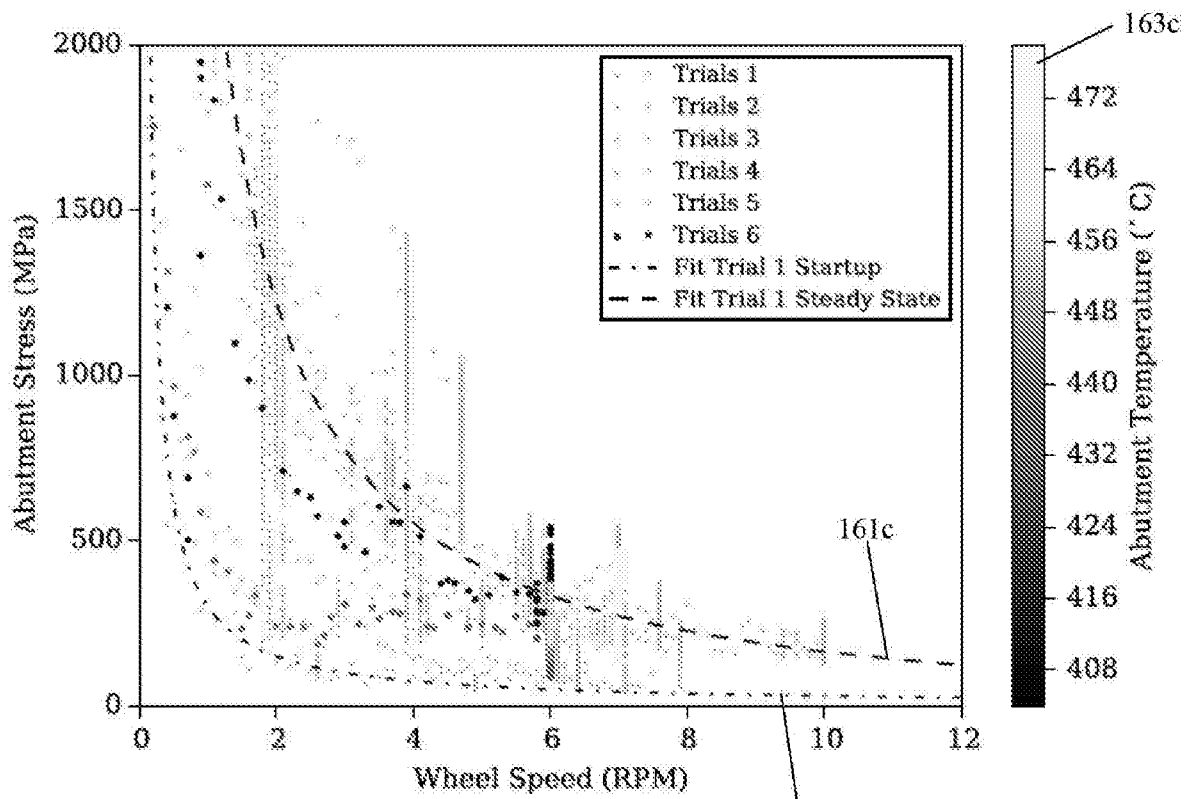


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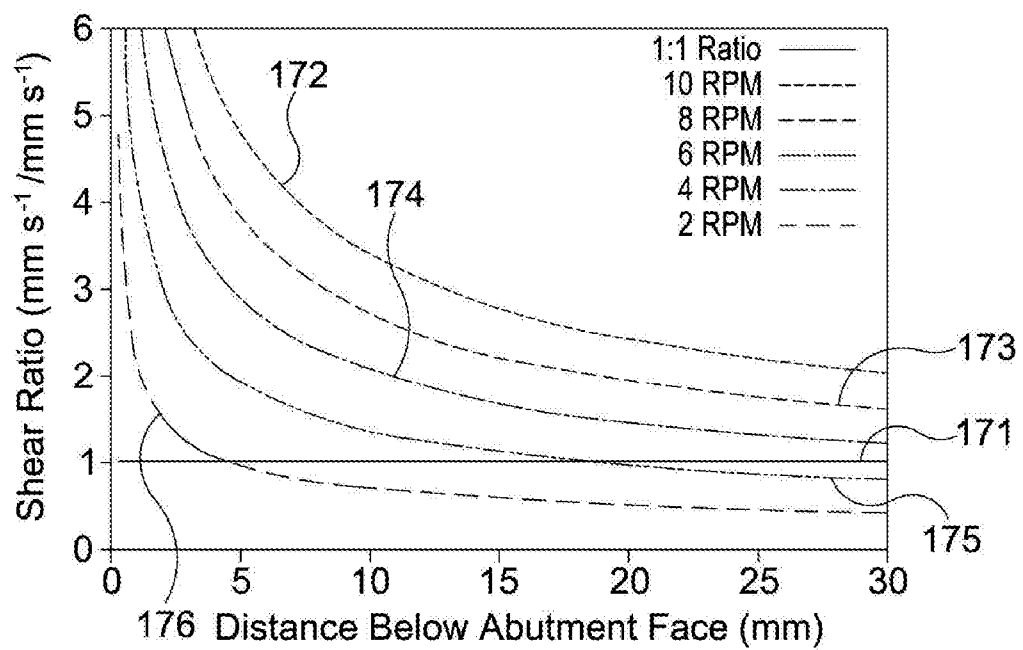
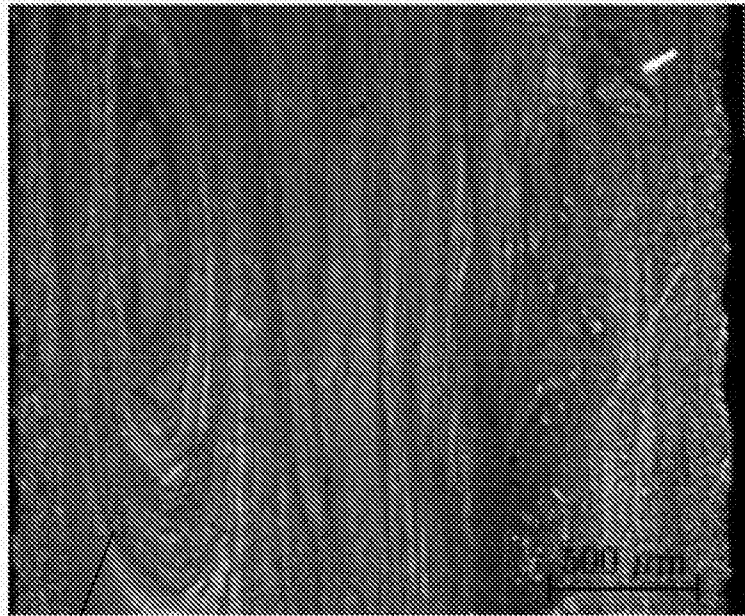


Figure 17

INCORPORATED BY REFERENCE (RULE 20.6)



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Figure 18



Figure 19

INCORPORATED BY REFERENCE (RULE 20.6)

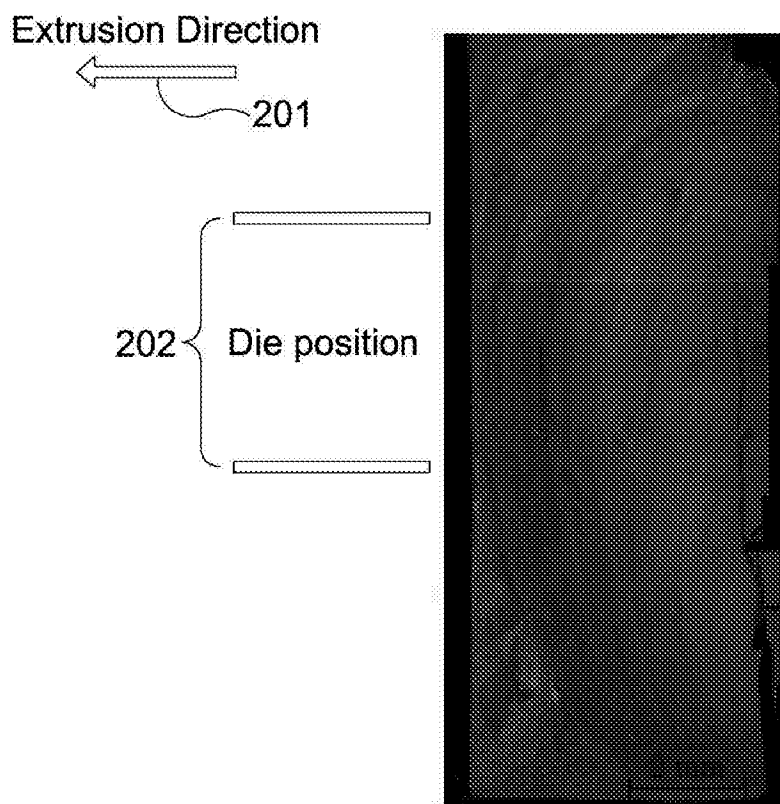


Figure 20

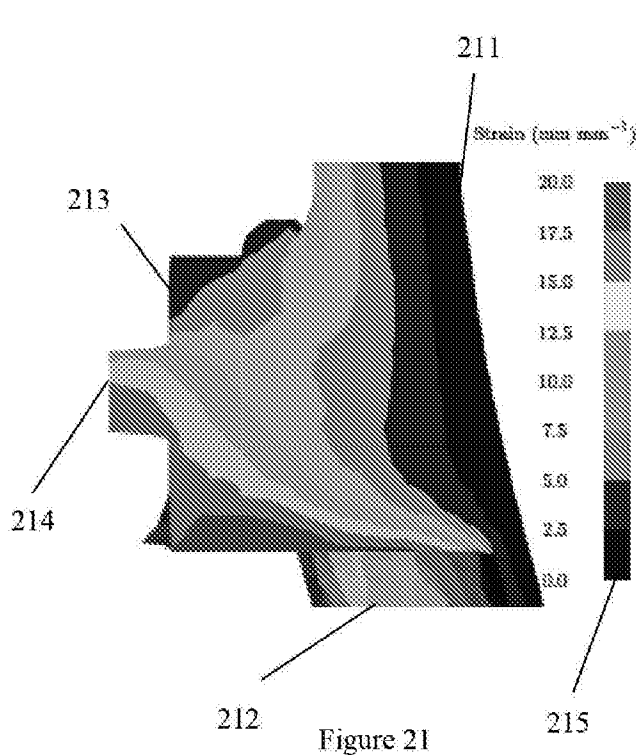


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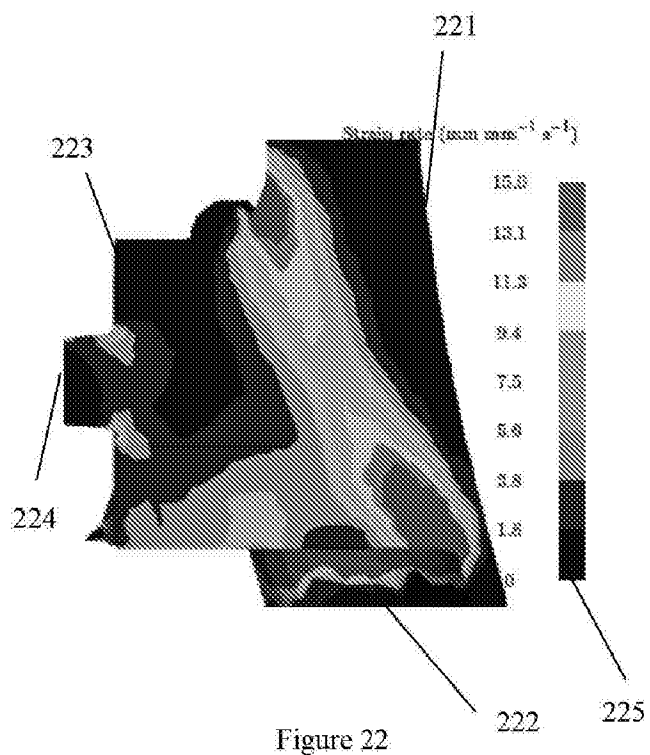


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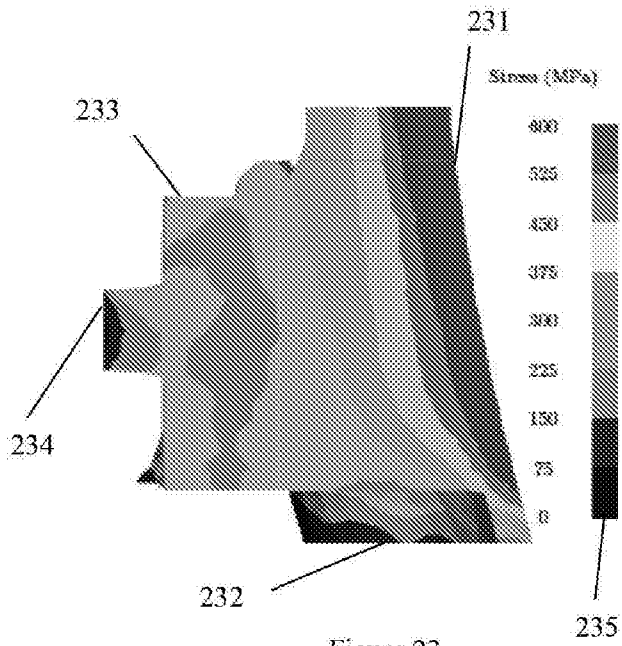


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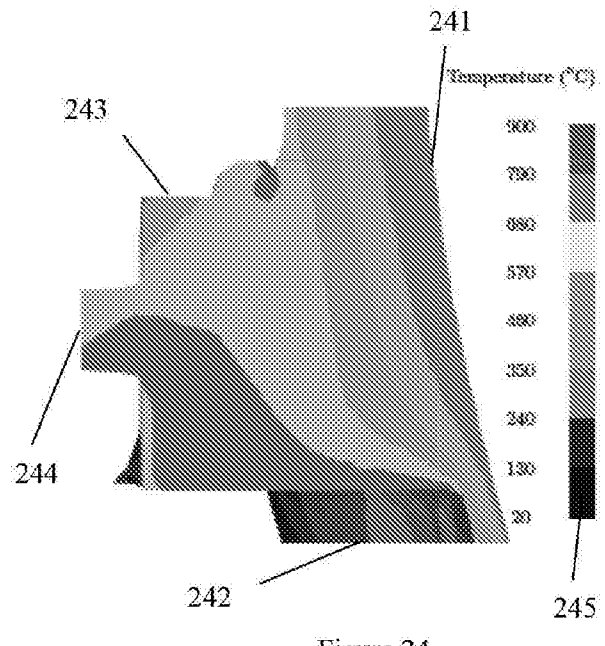


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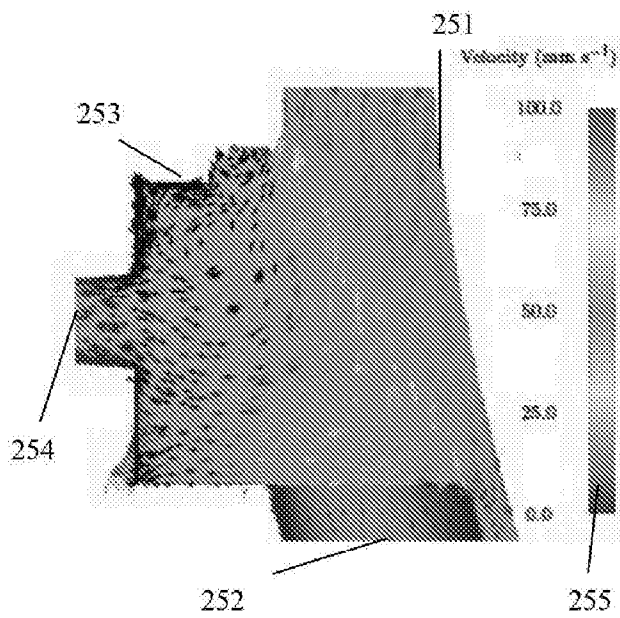


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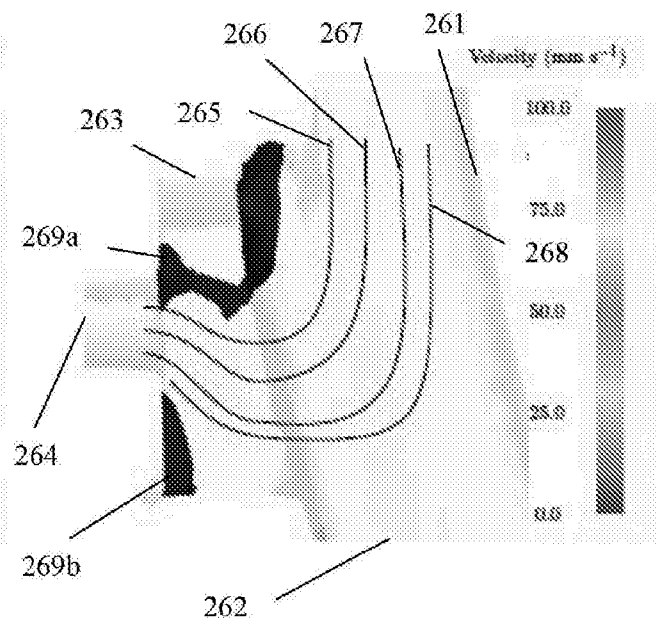


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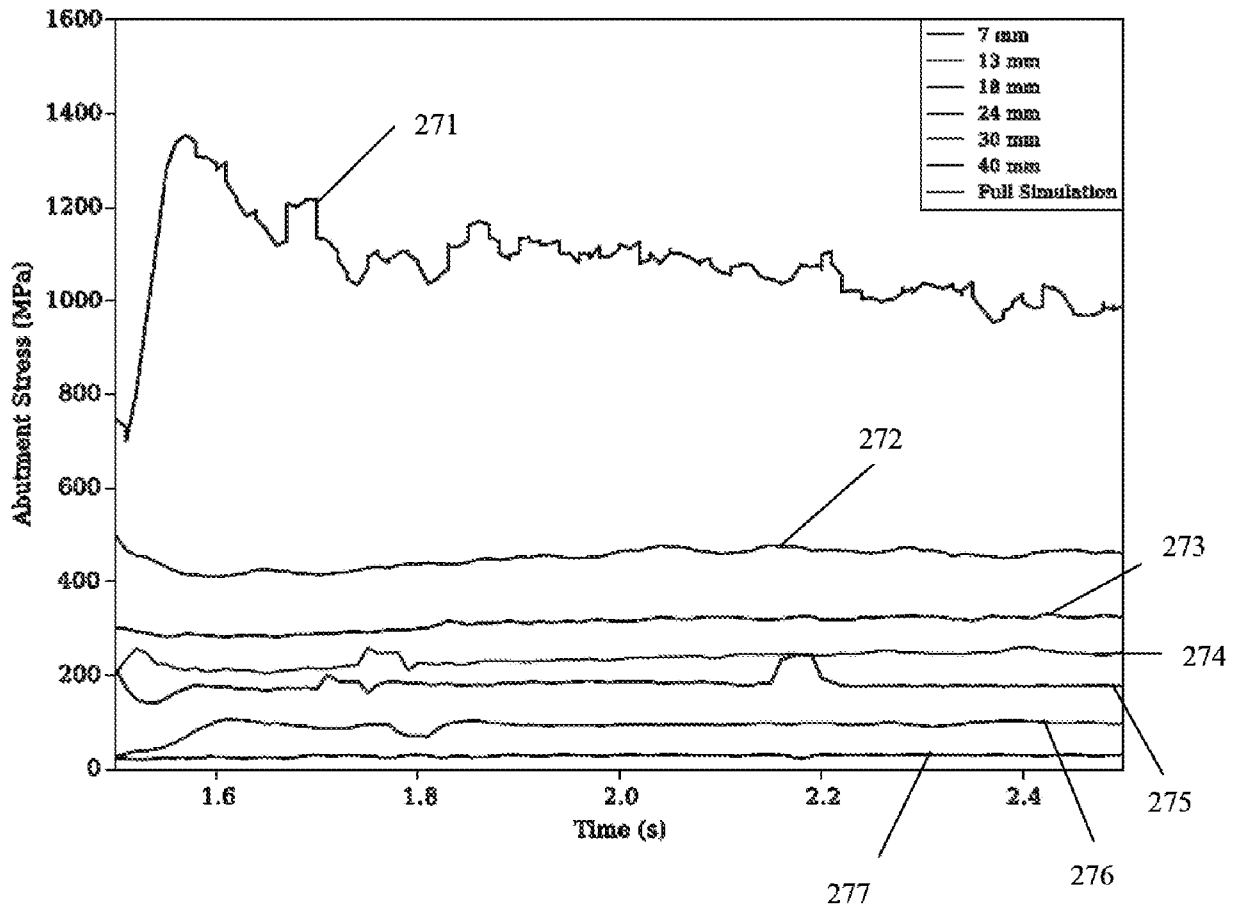


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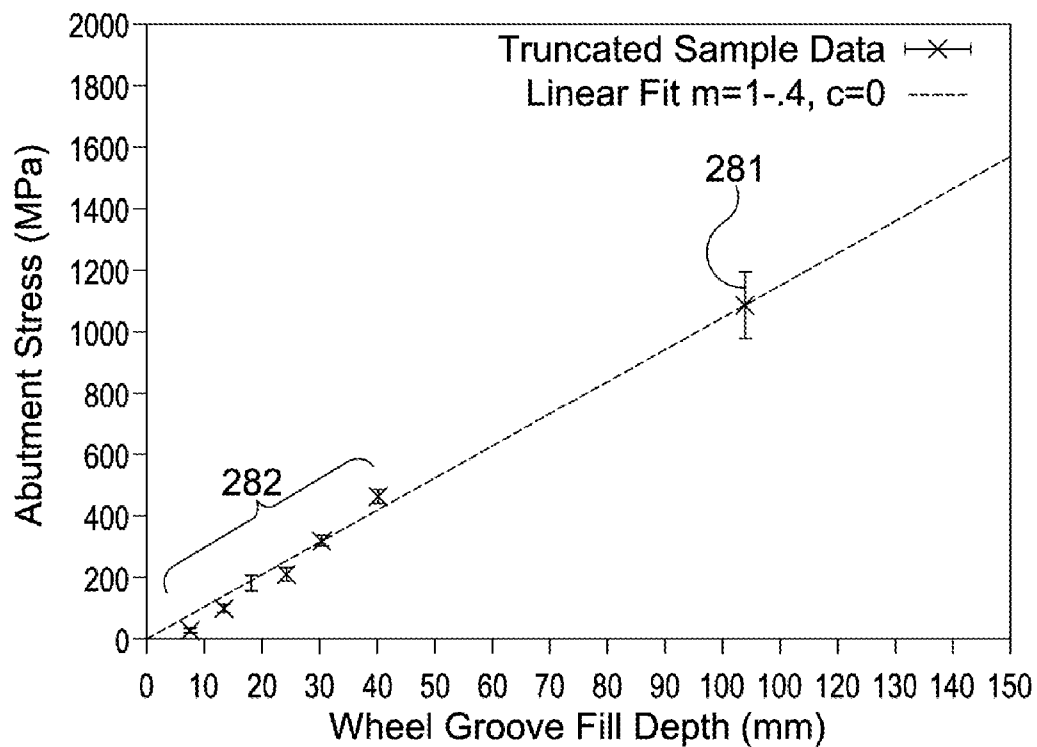


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INCORPORATED BY REFERENCE (RULE 20.6)

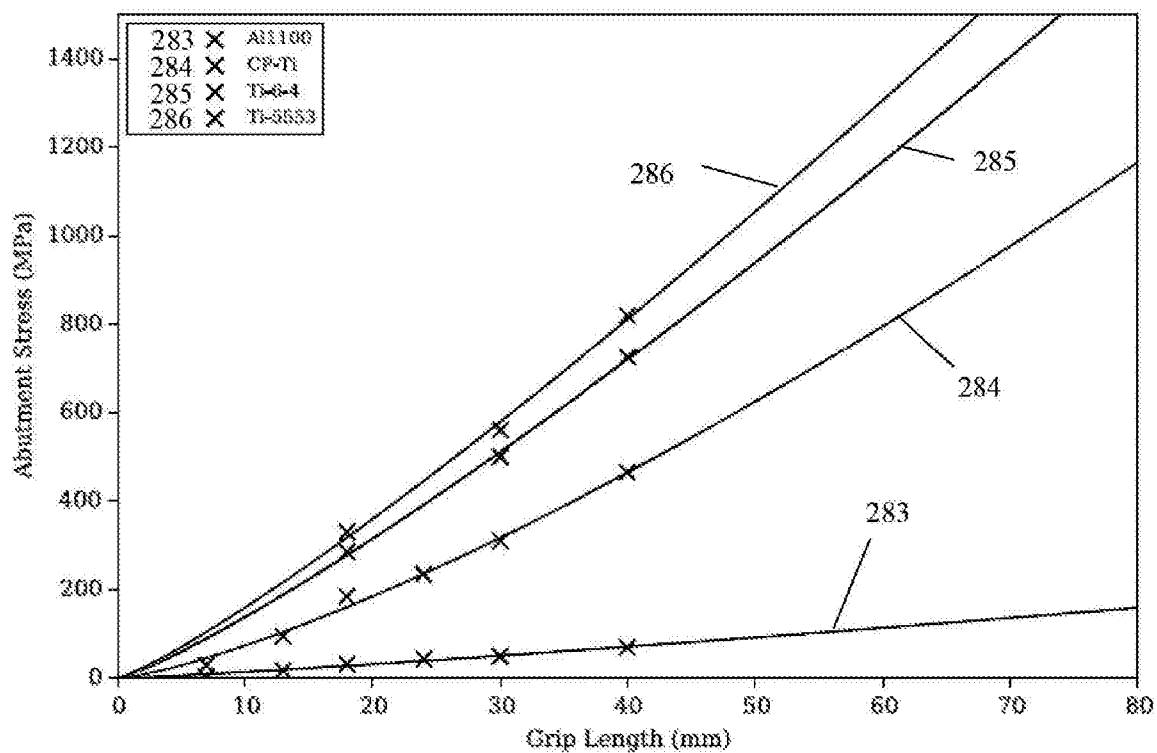


Figure 28B

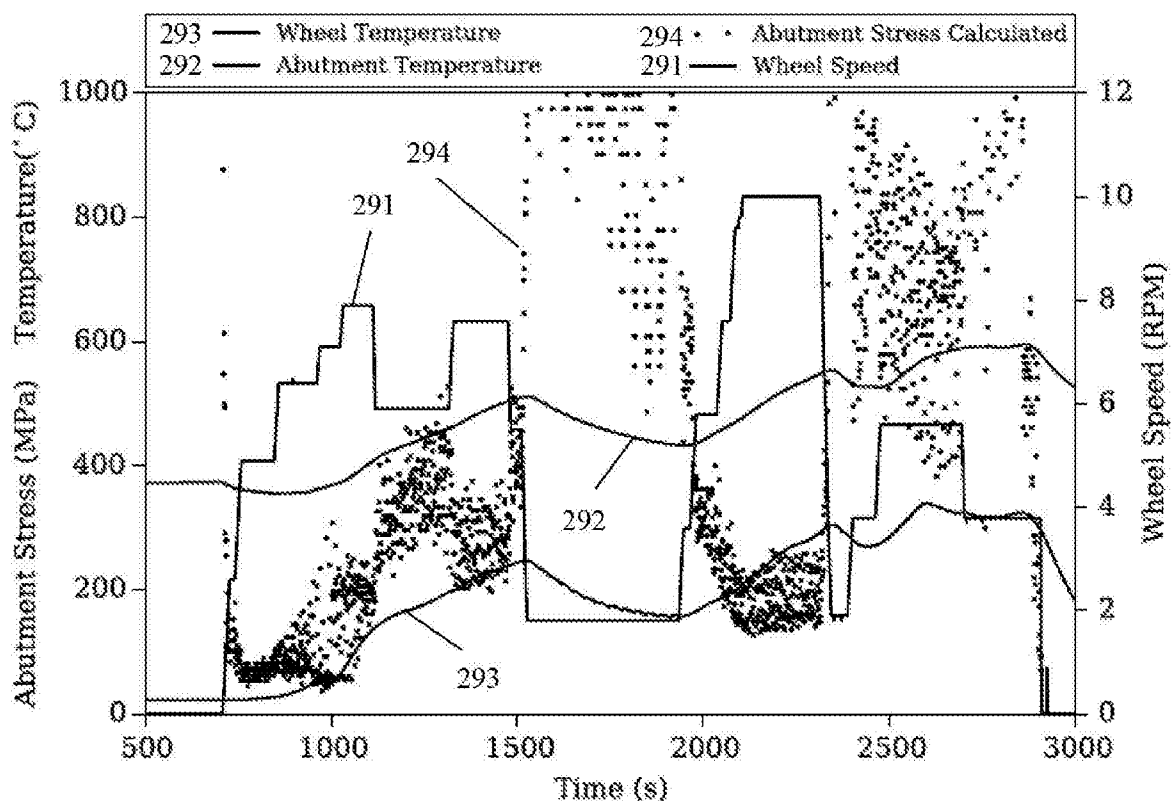


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INCORPORATED BY REFERENCE (RULE 20.6)

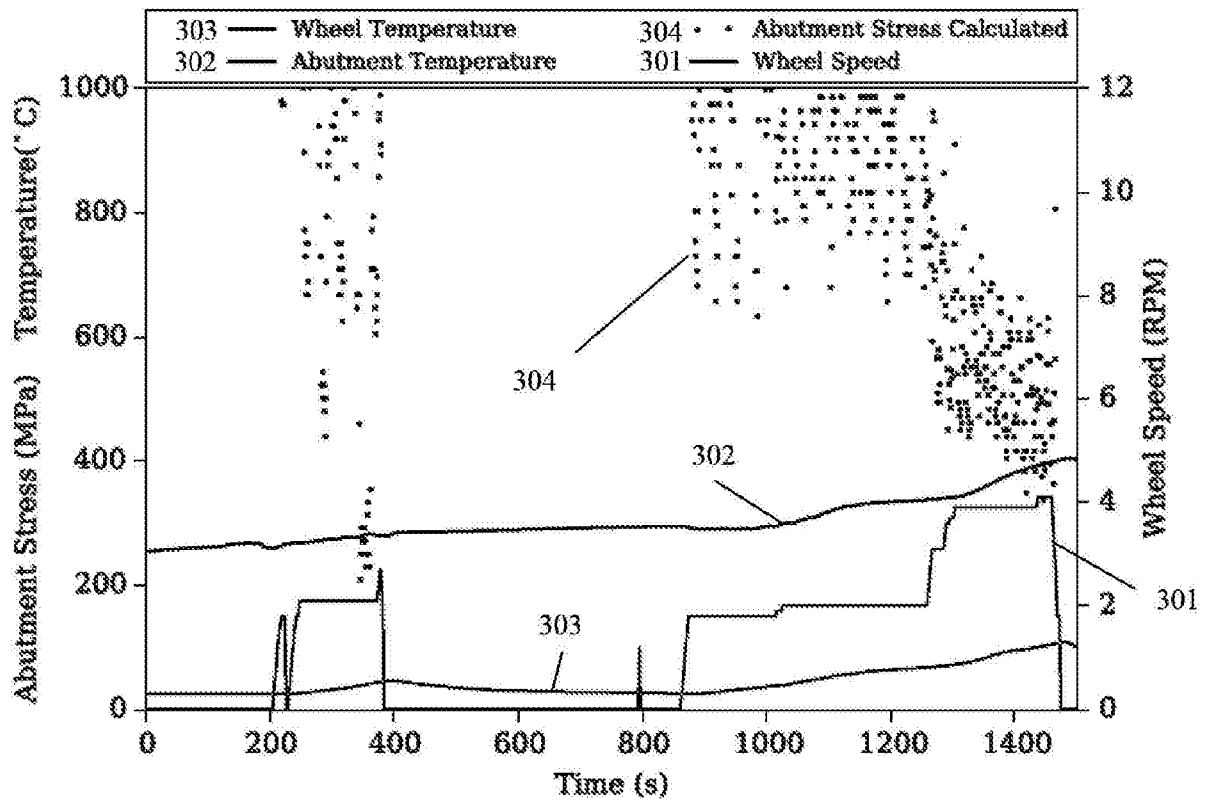


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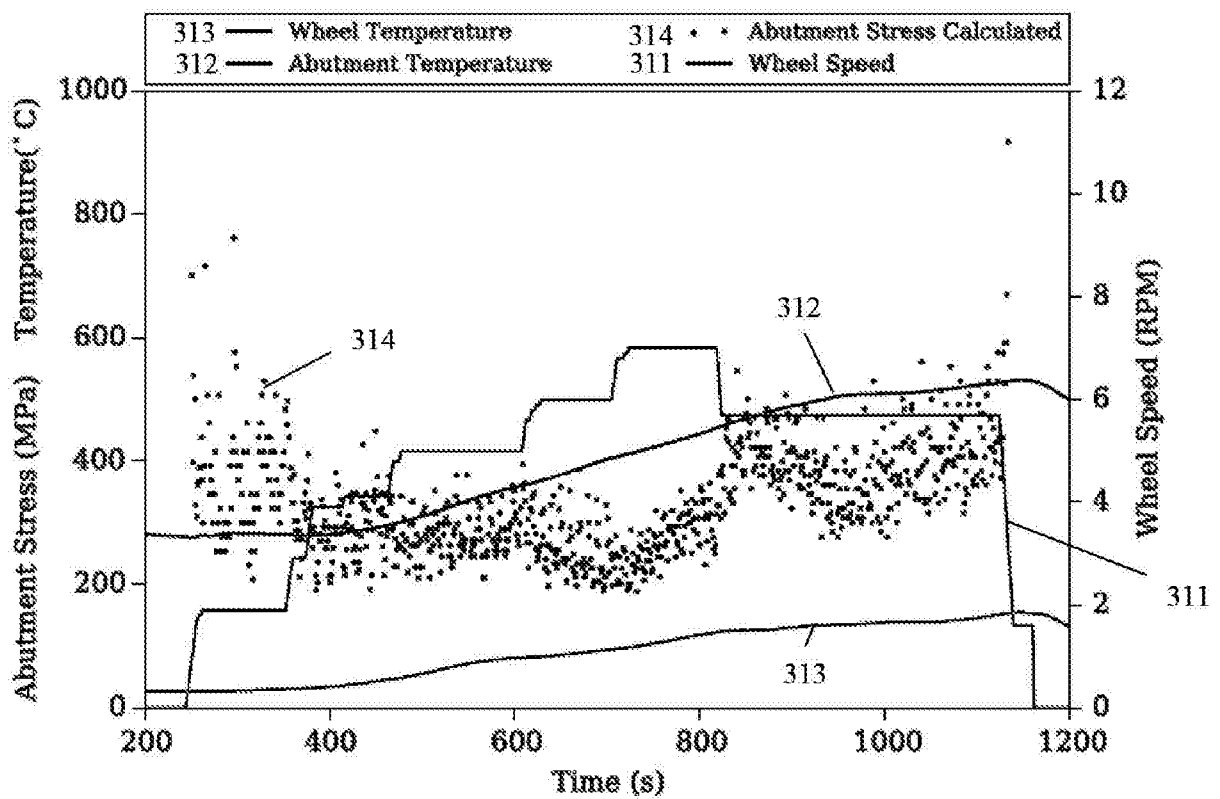


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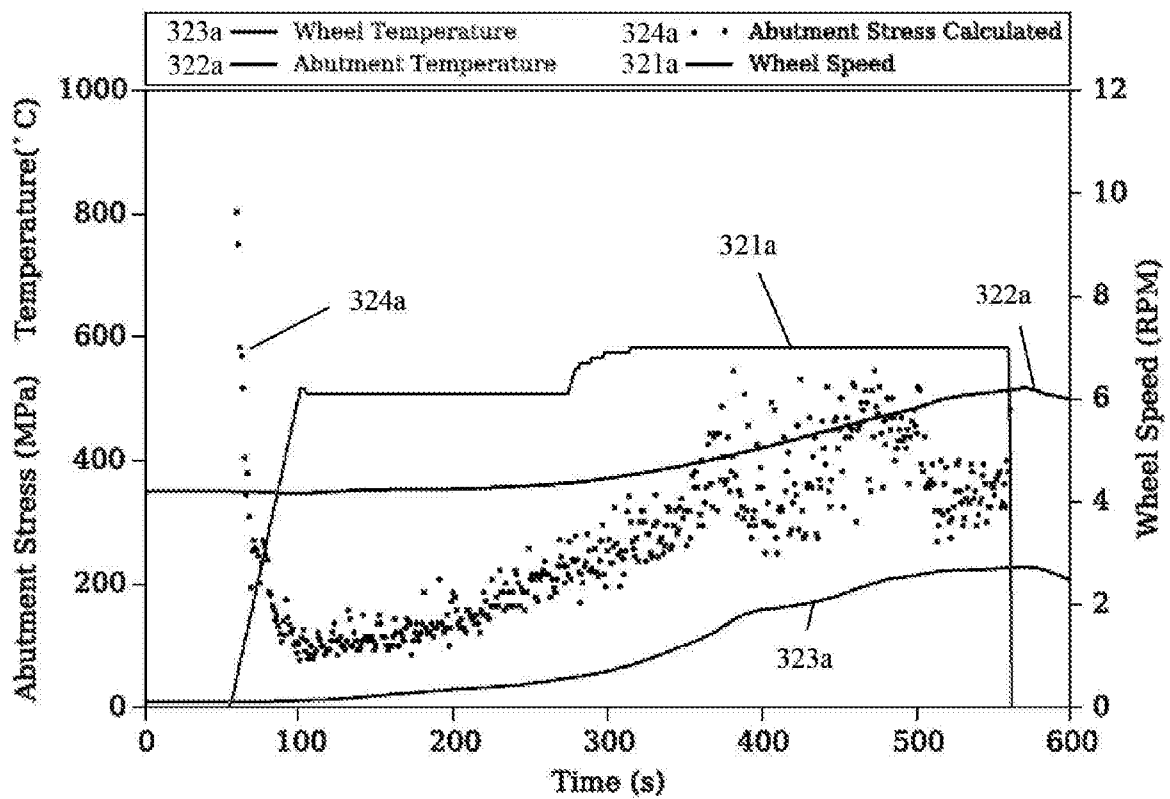


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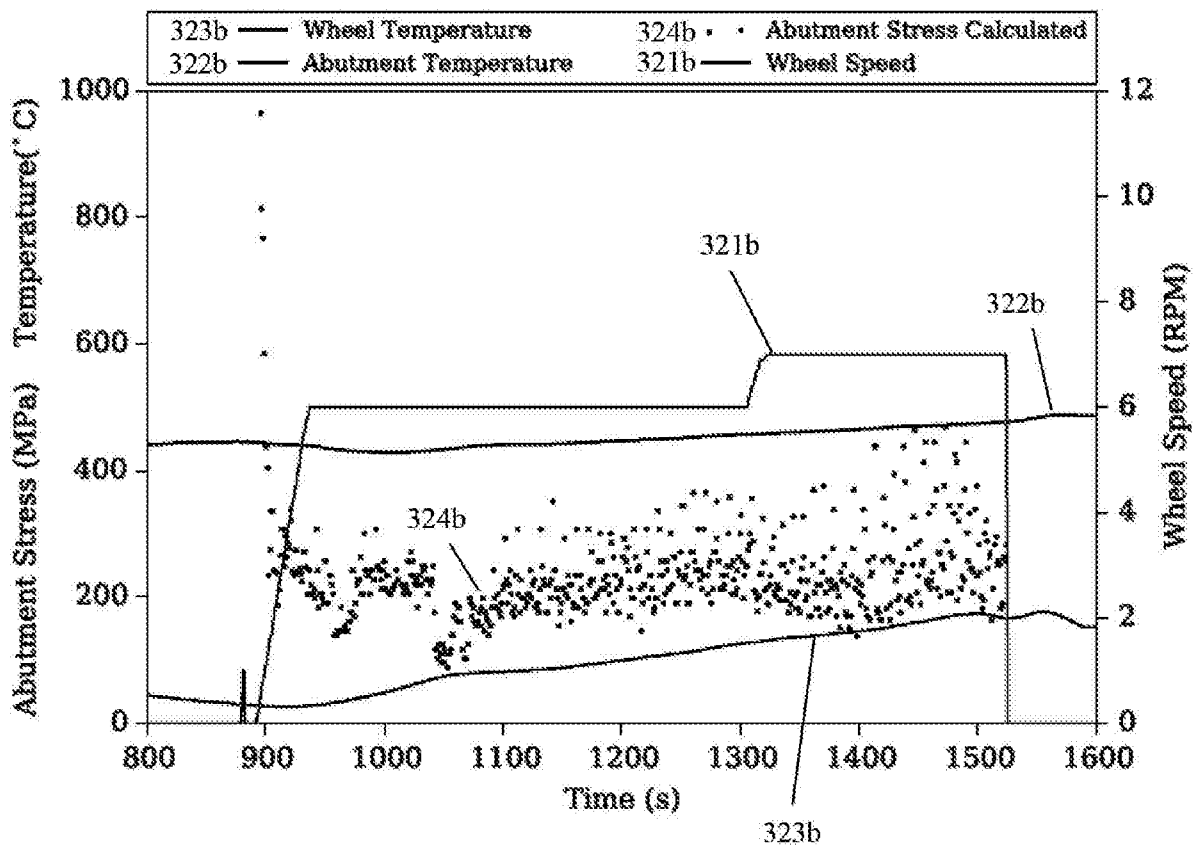


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INCORPORATED BY REFERENCE (RULE 20.6)

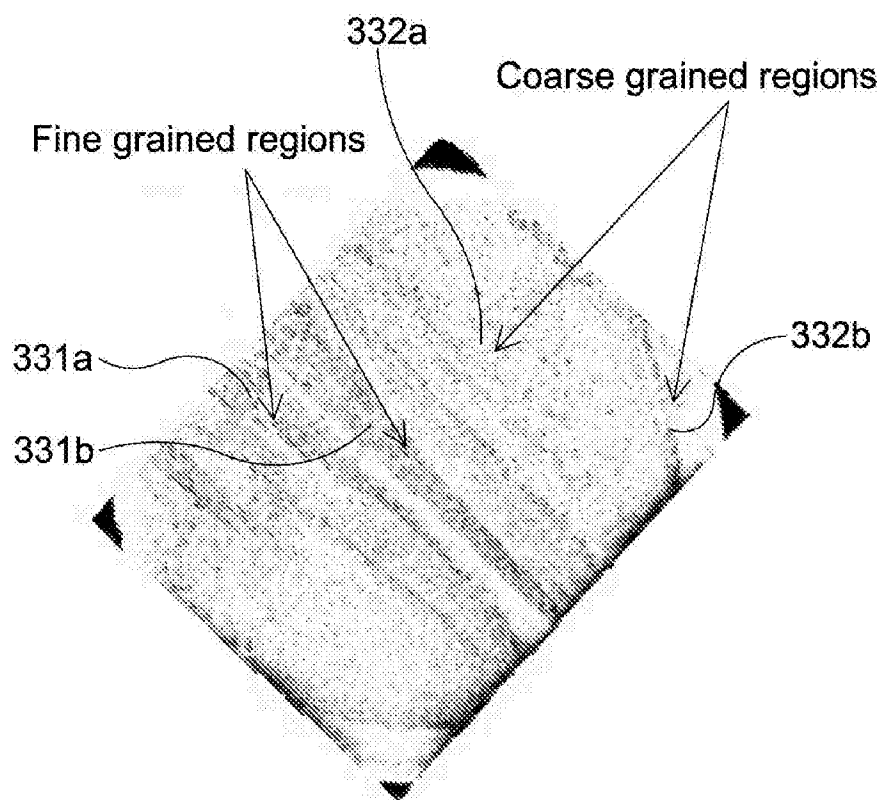


Figure 33



Figure 34

INCORPORATED BY REFERENCE (RULE 20.6)

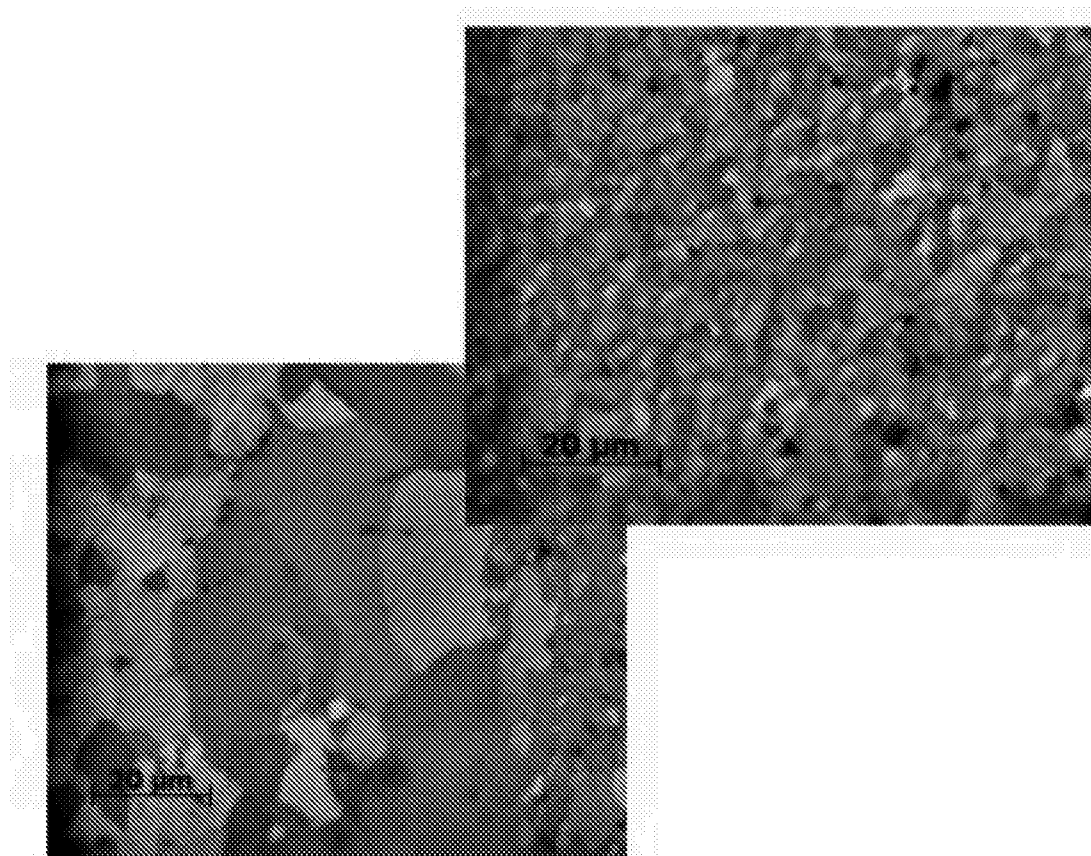


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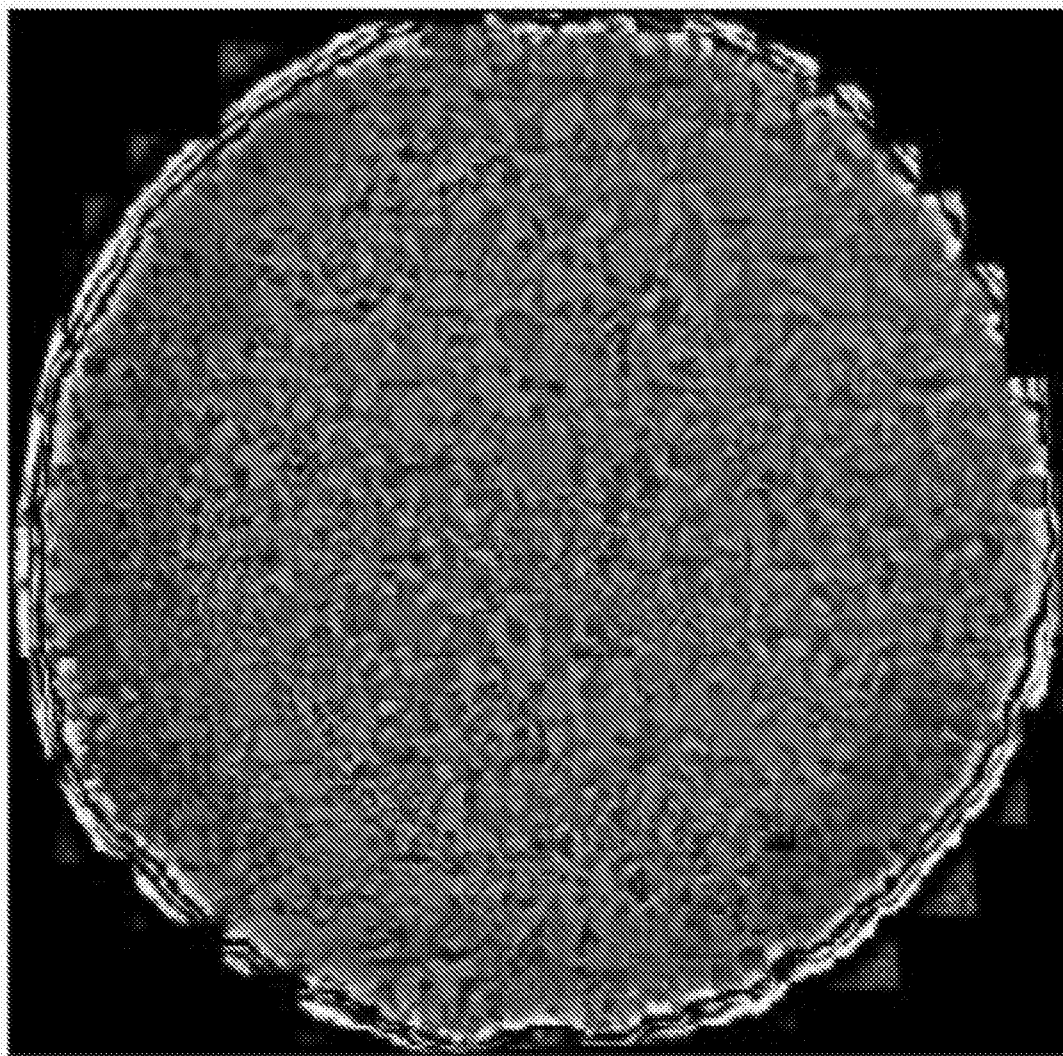


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INCORPORATED BY REFERENCE (RULE 20.6)

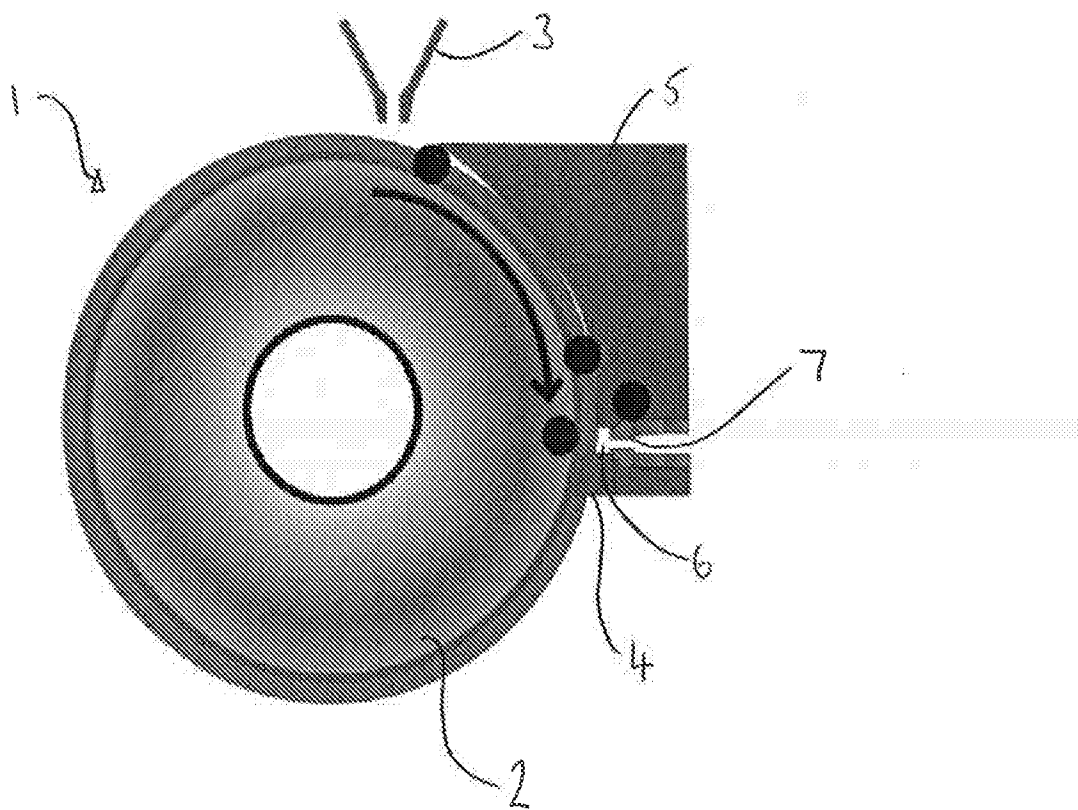


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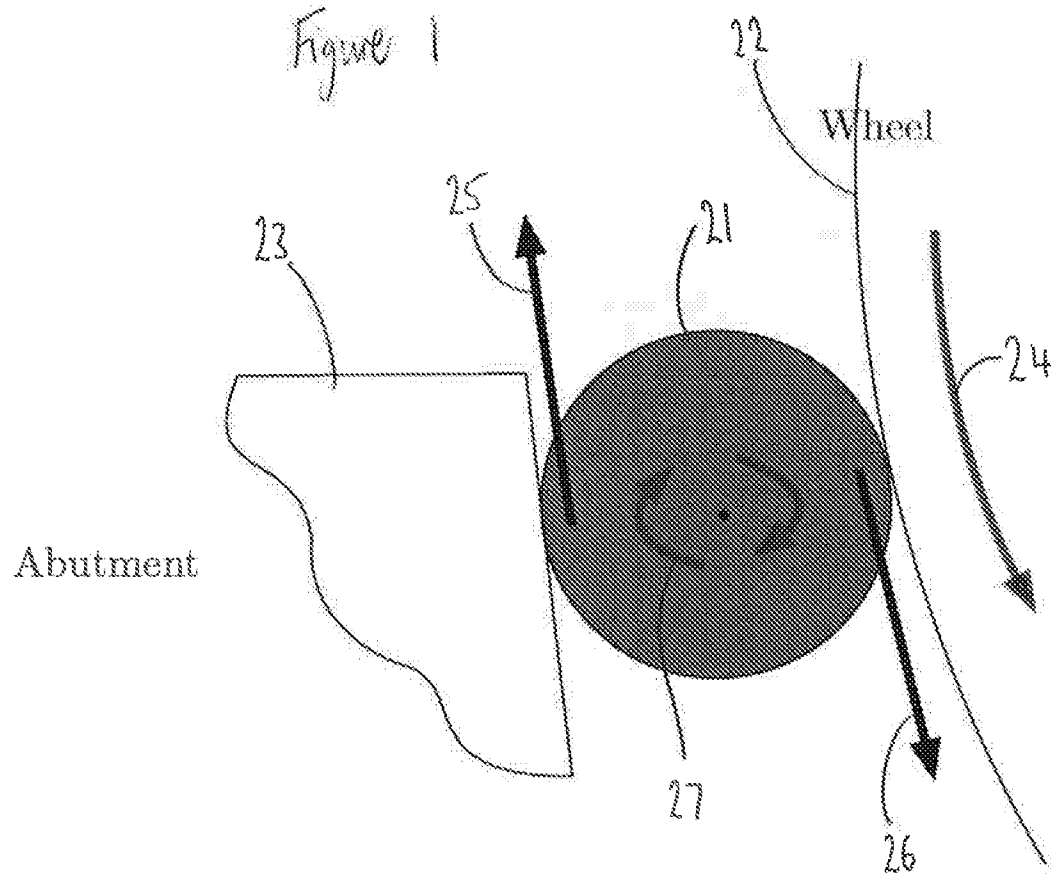


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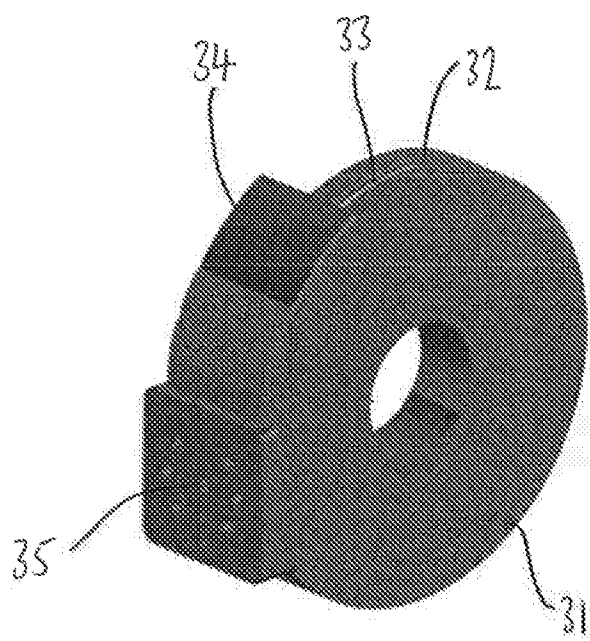


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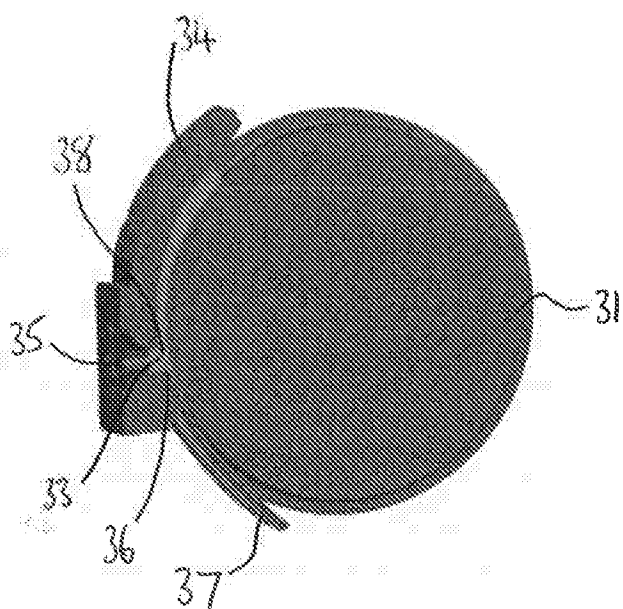


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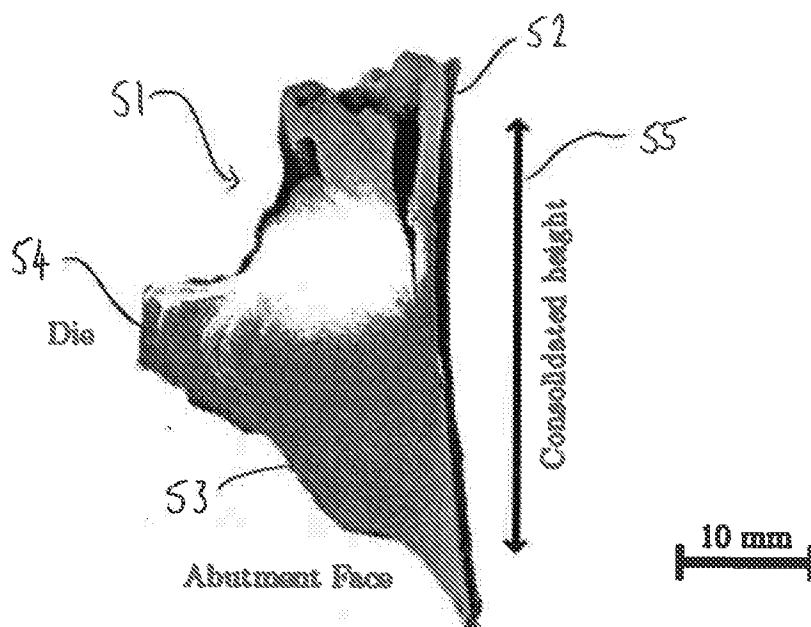


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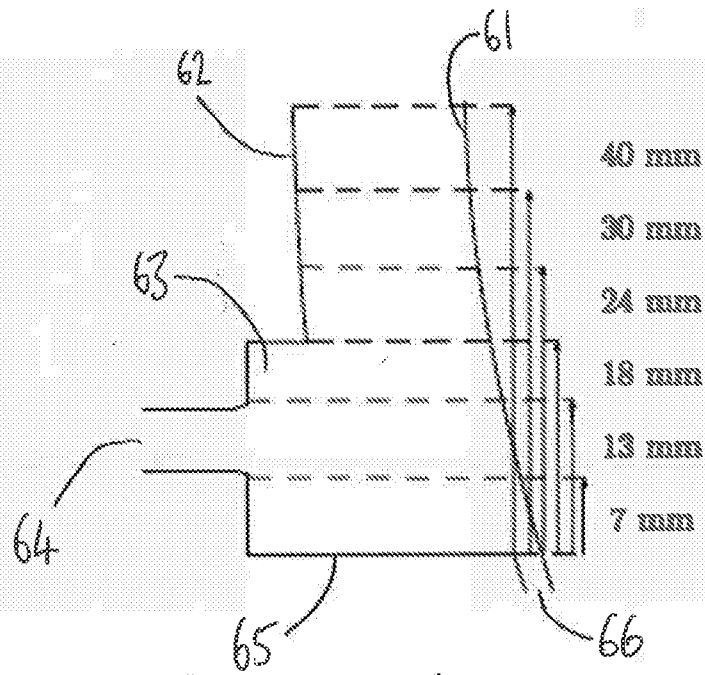


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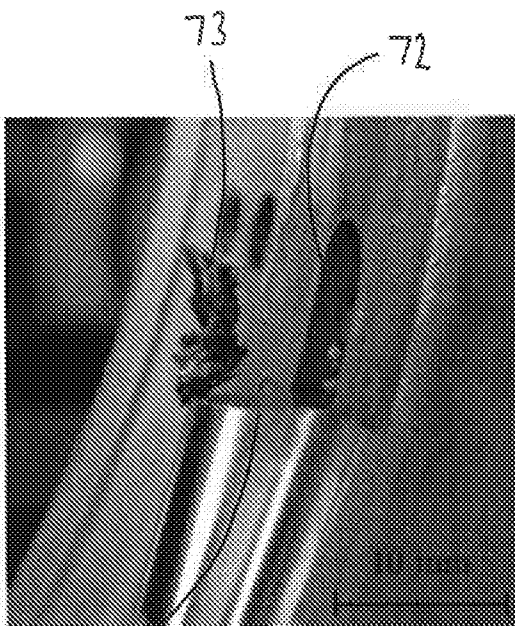


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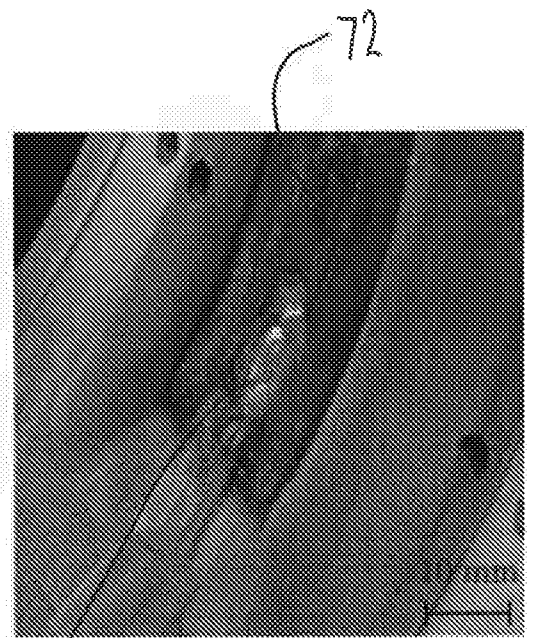


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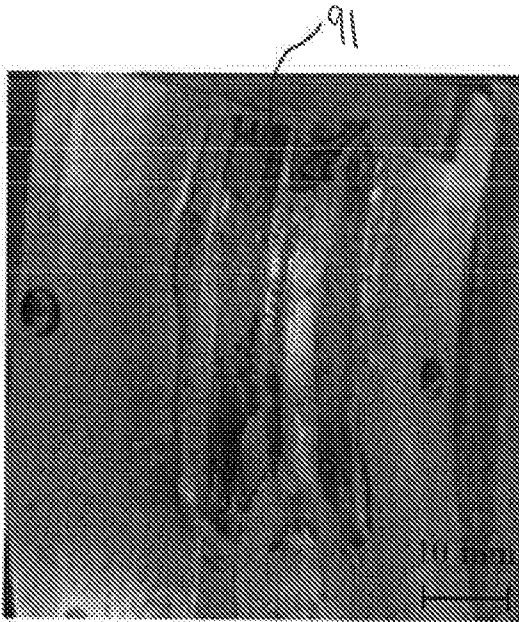


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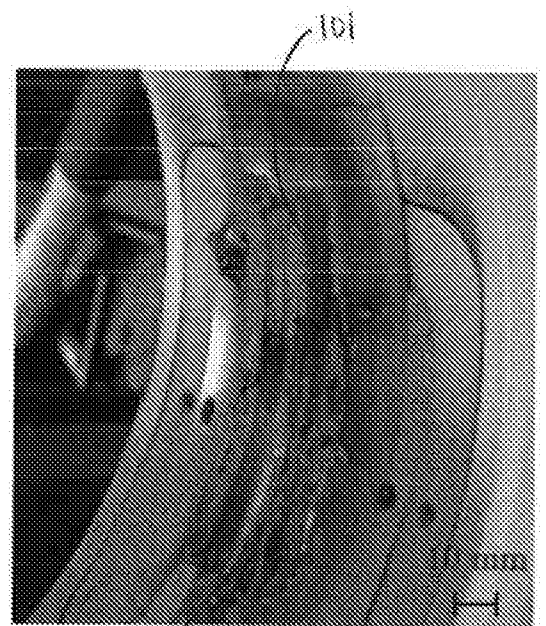


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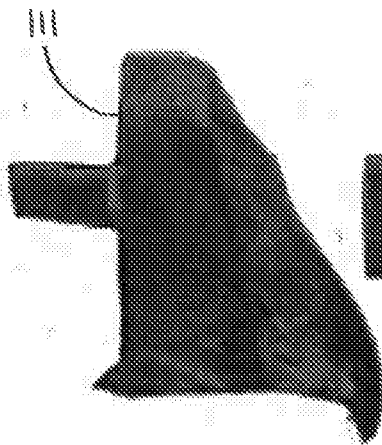


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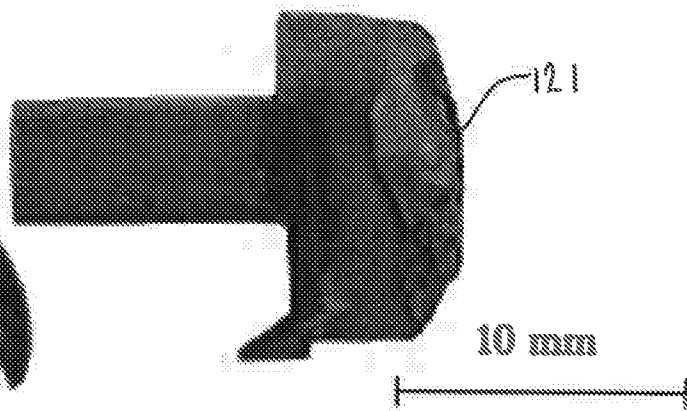


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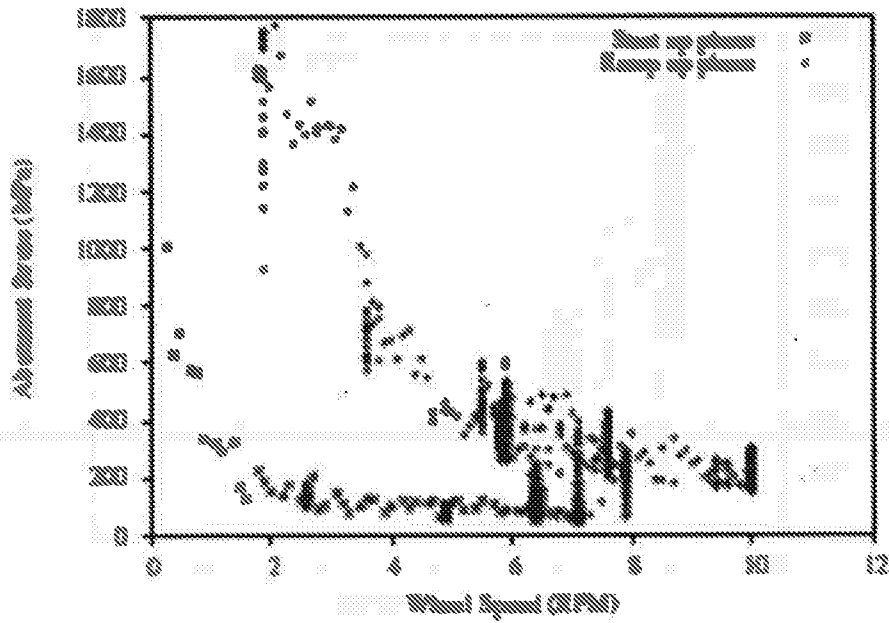


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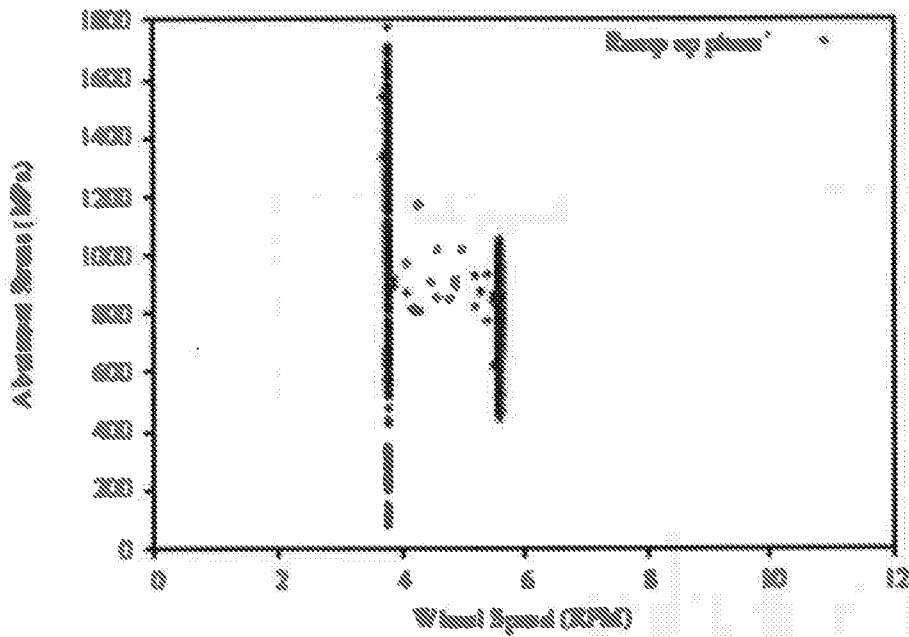


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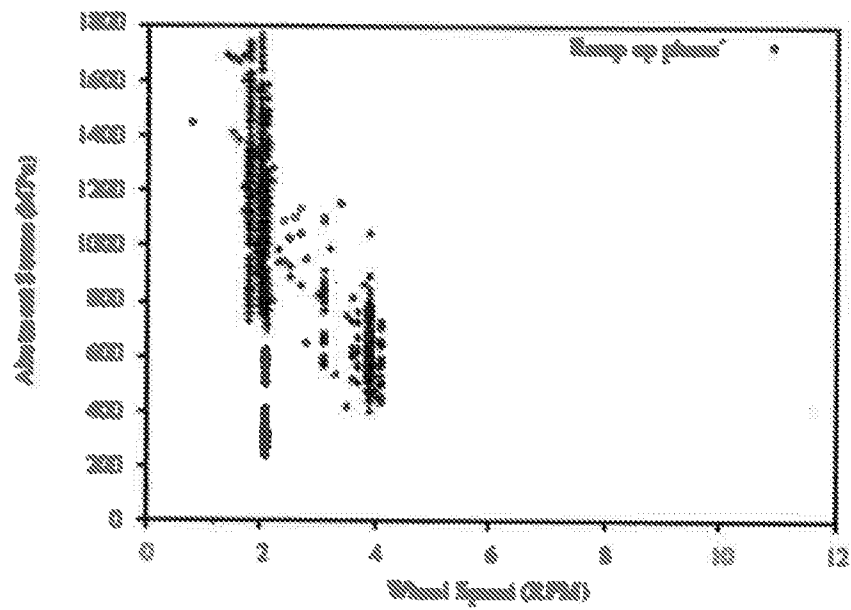


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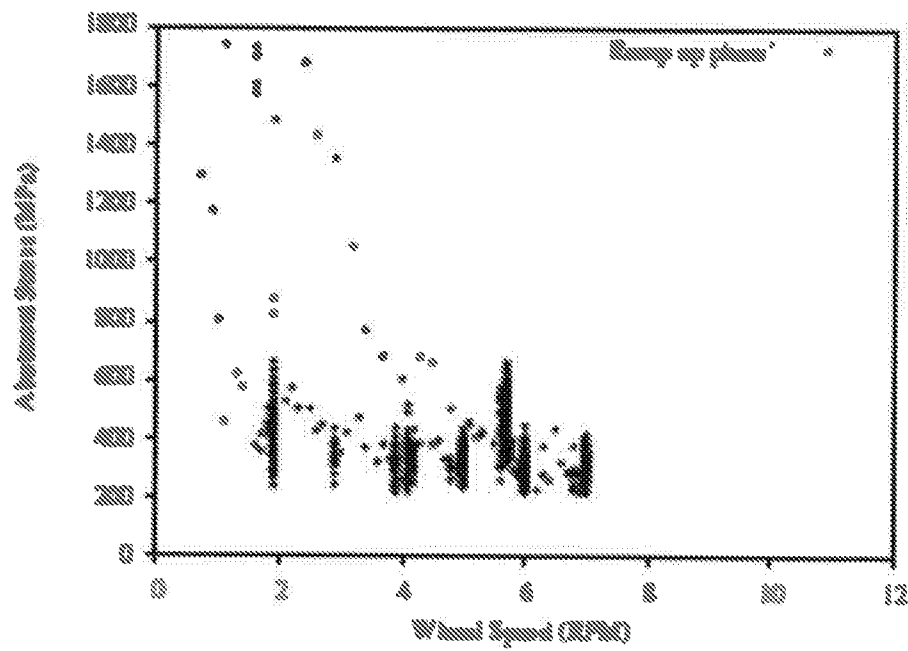


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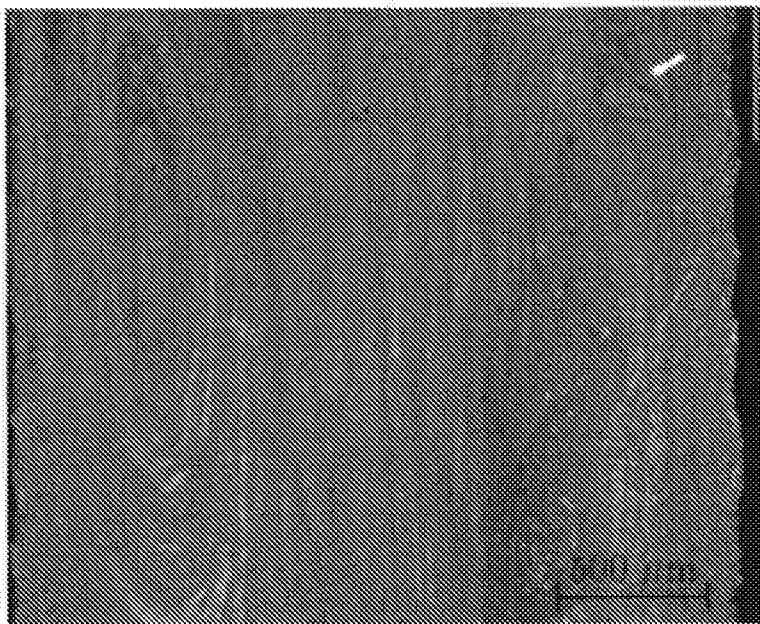
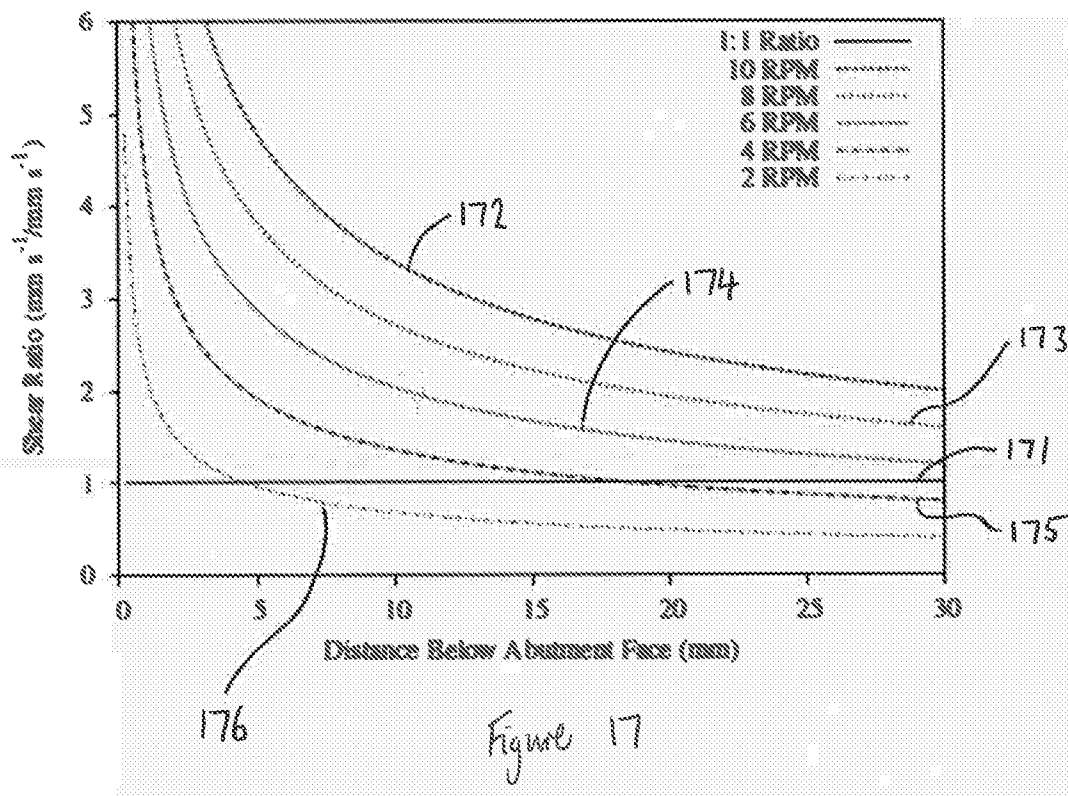


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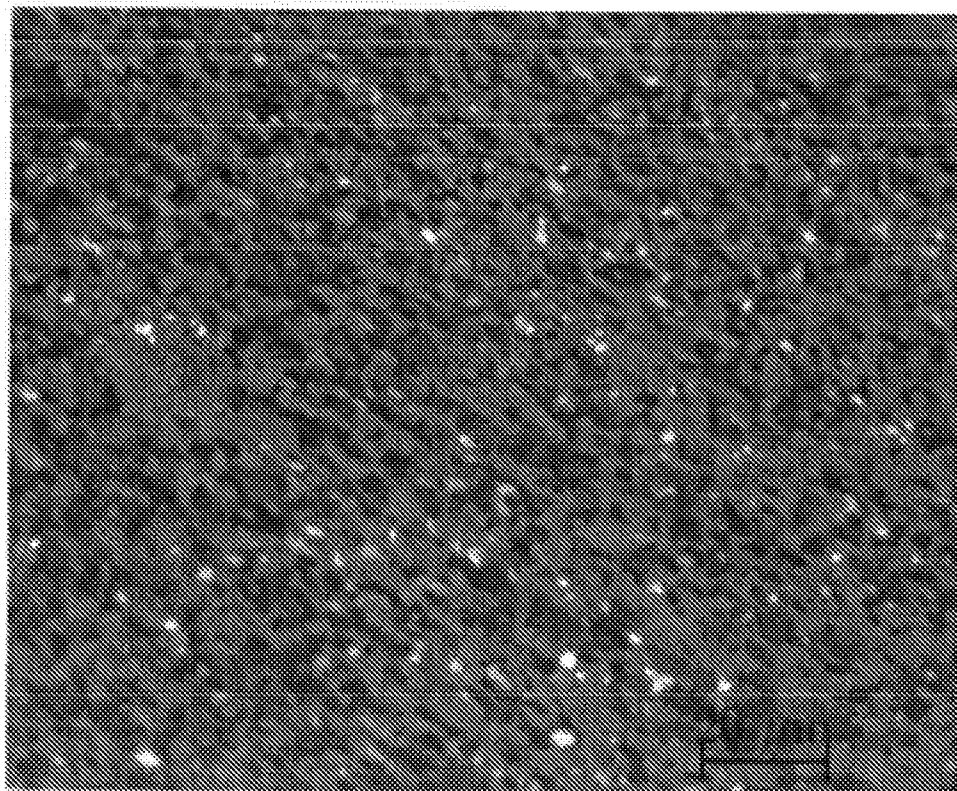


Figure 19

Extrusion Direction



201

202

Die position

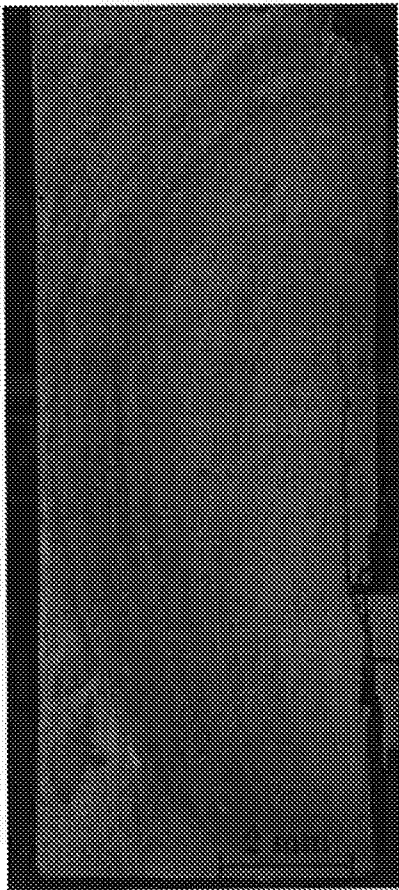
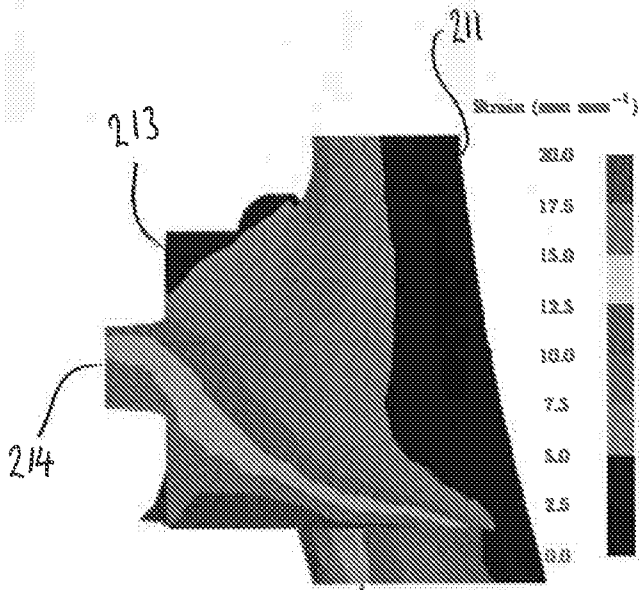
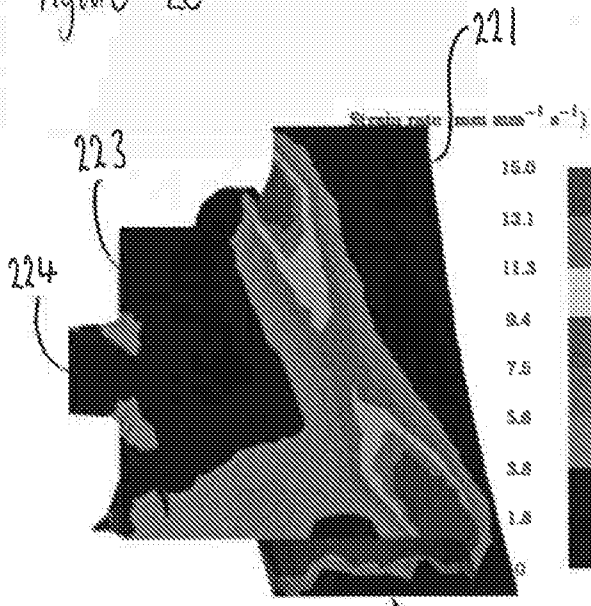


Figure 20



212

Figure 21



212

Figure 22

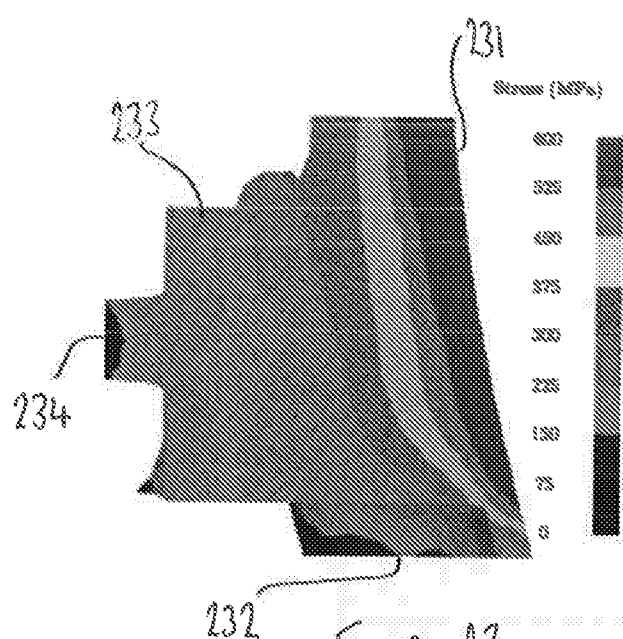


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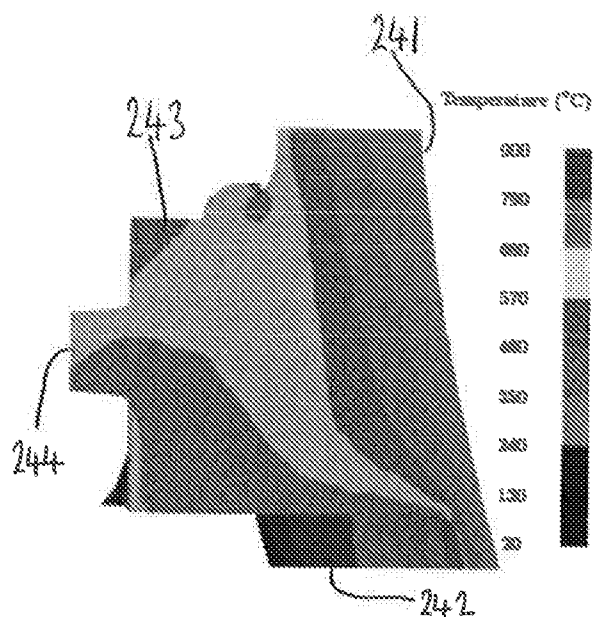


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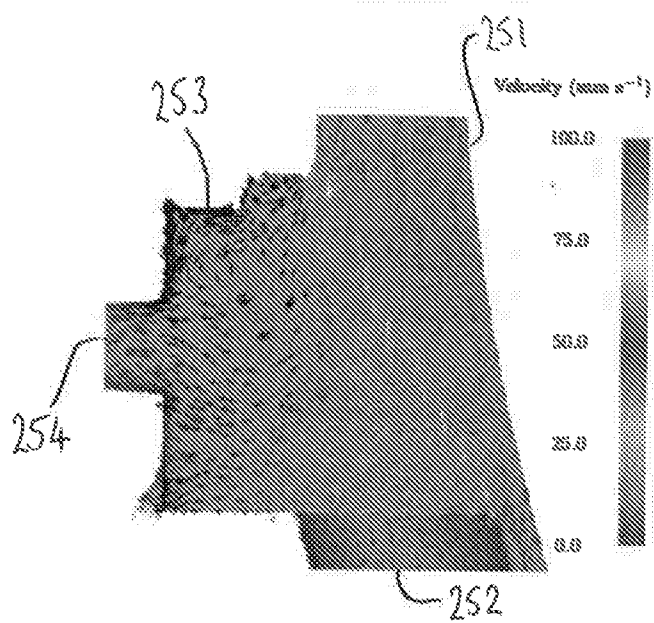


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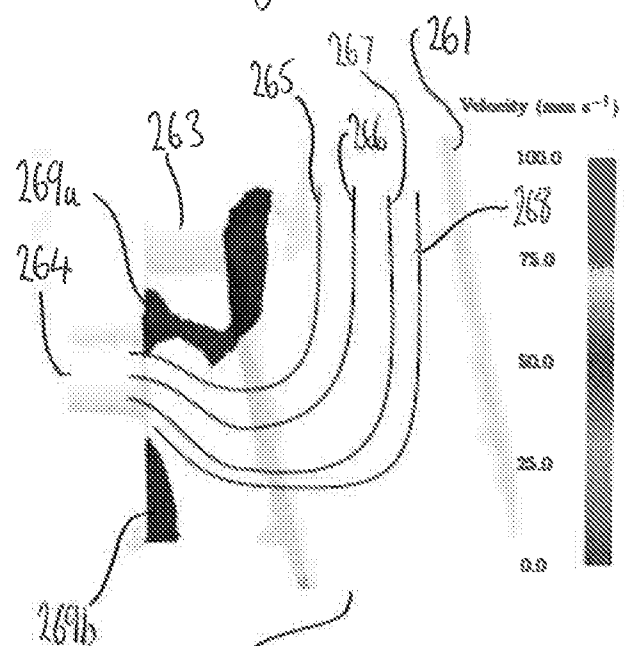


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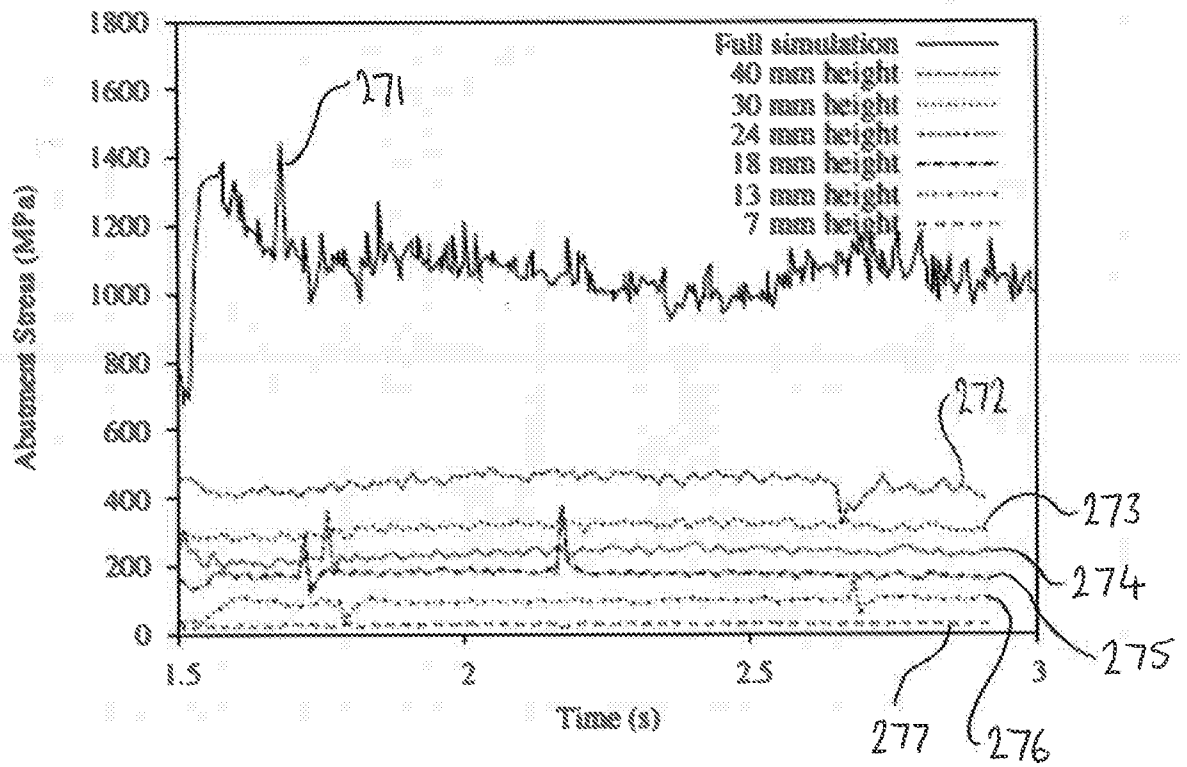


Figure 27

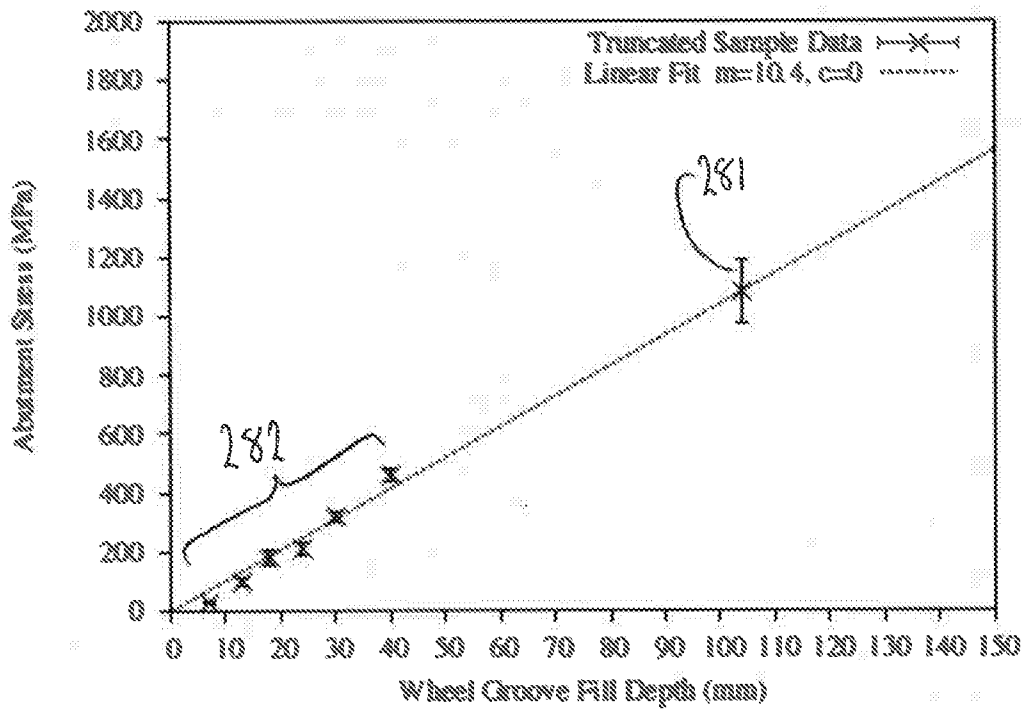


Figure 28

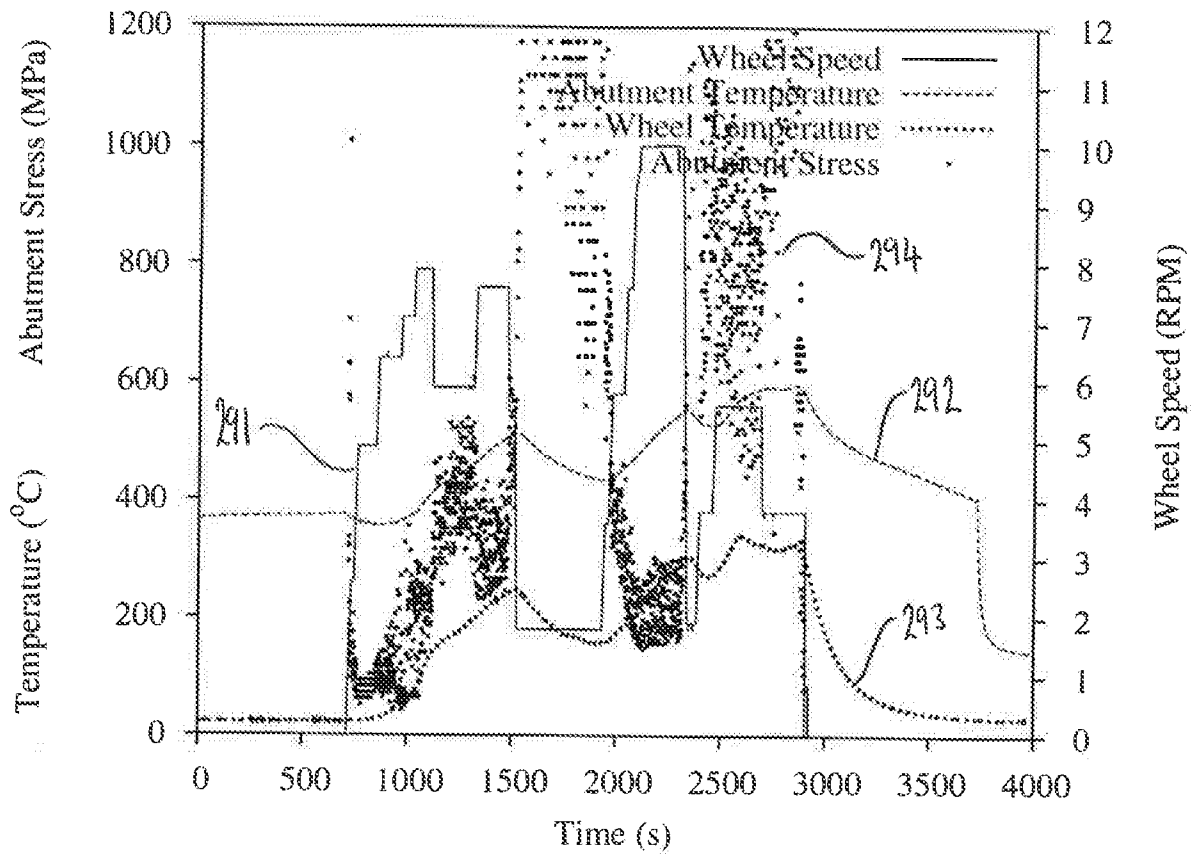


Figure 29

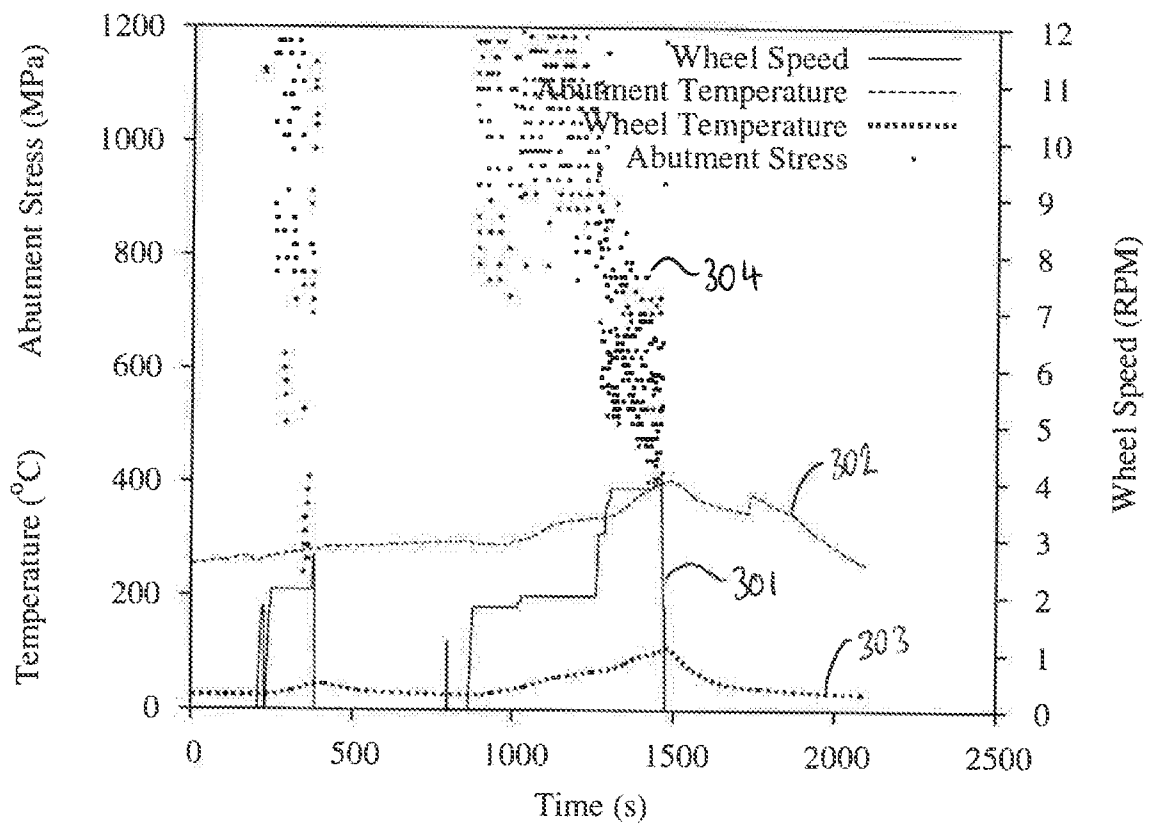


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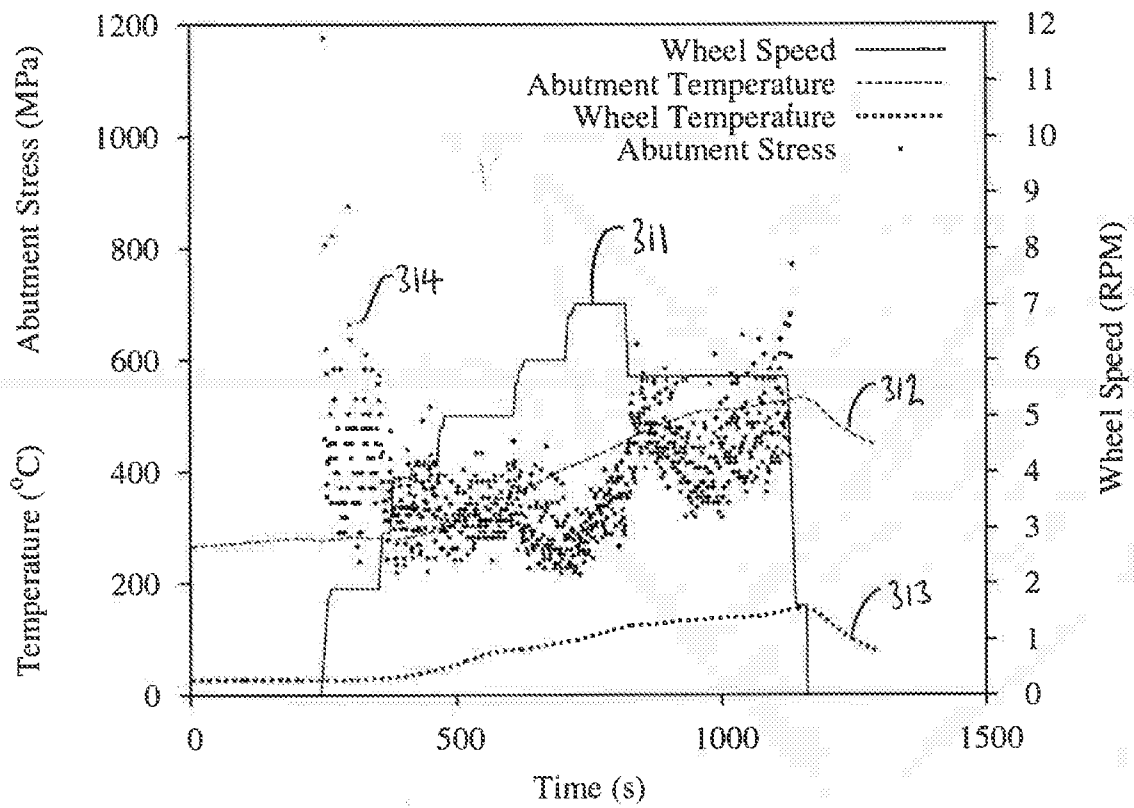


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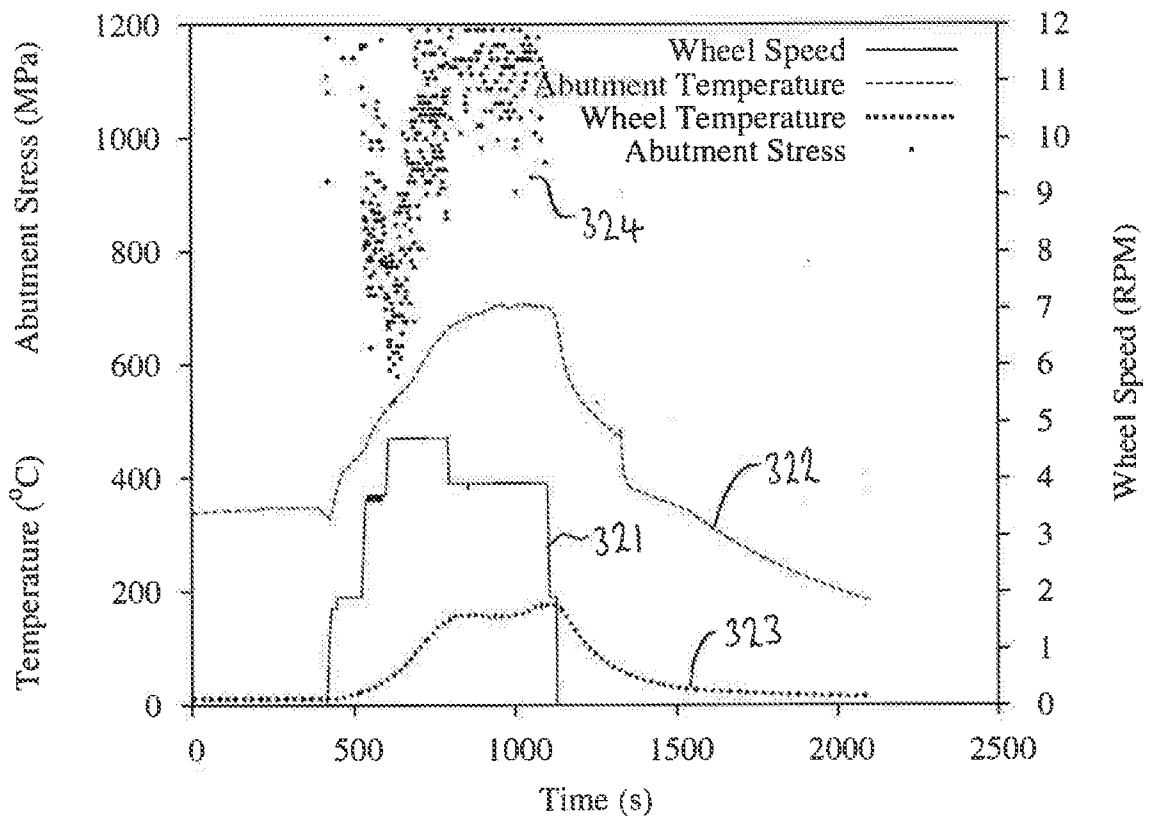


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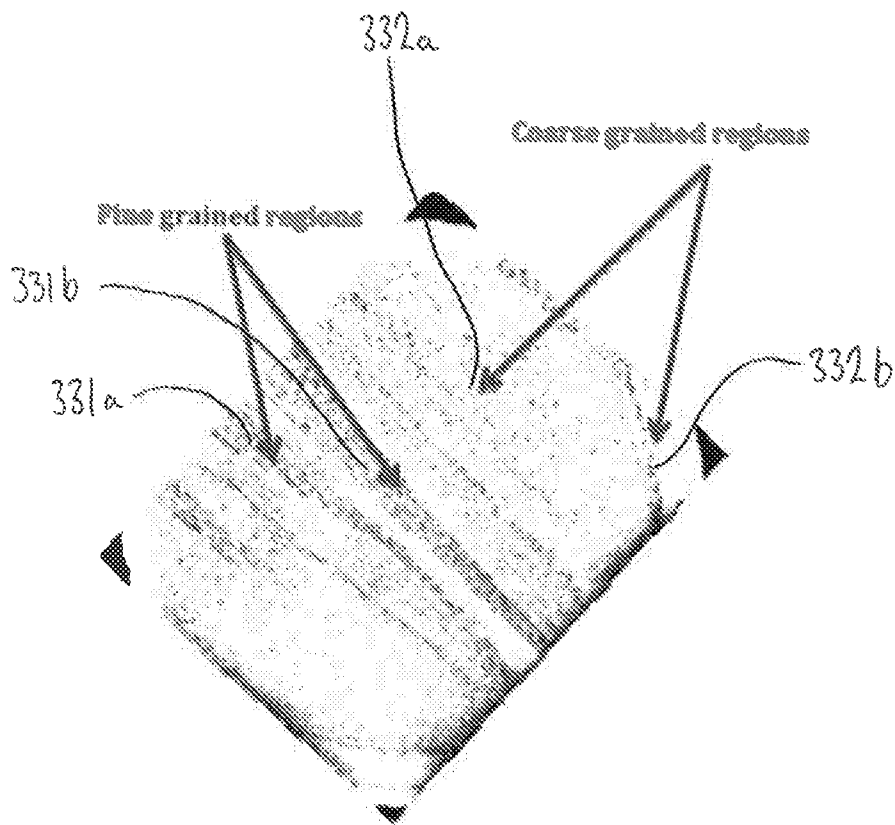


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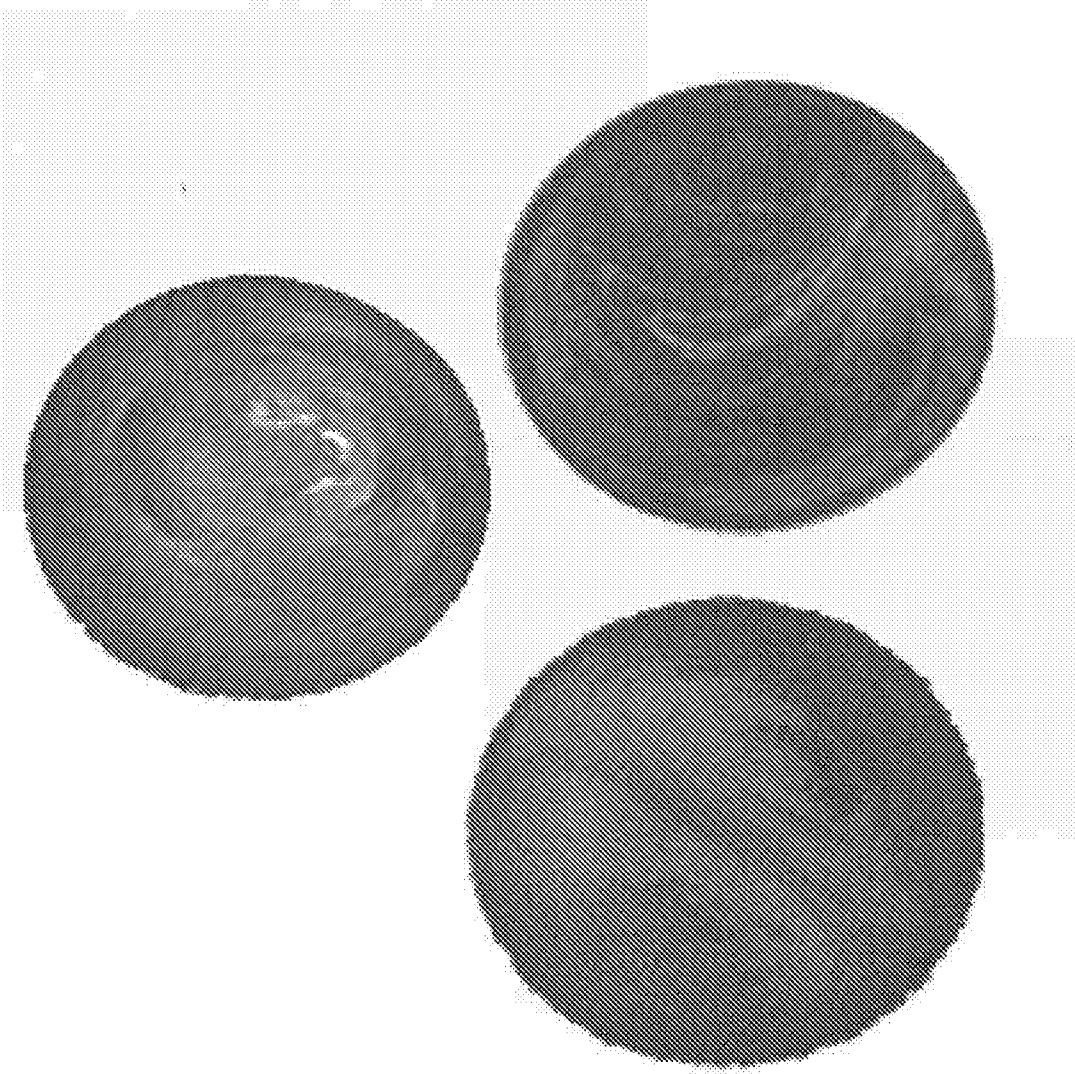


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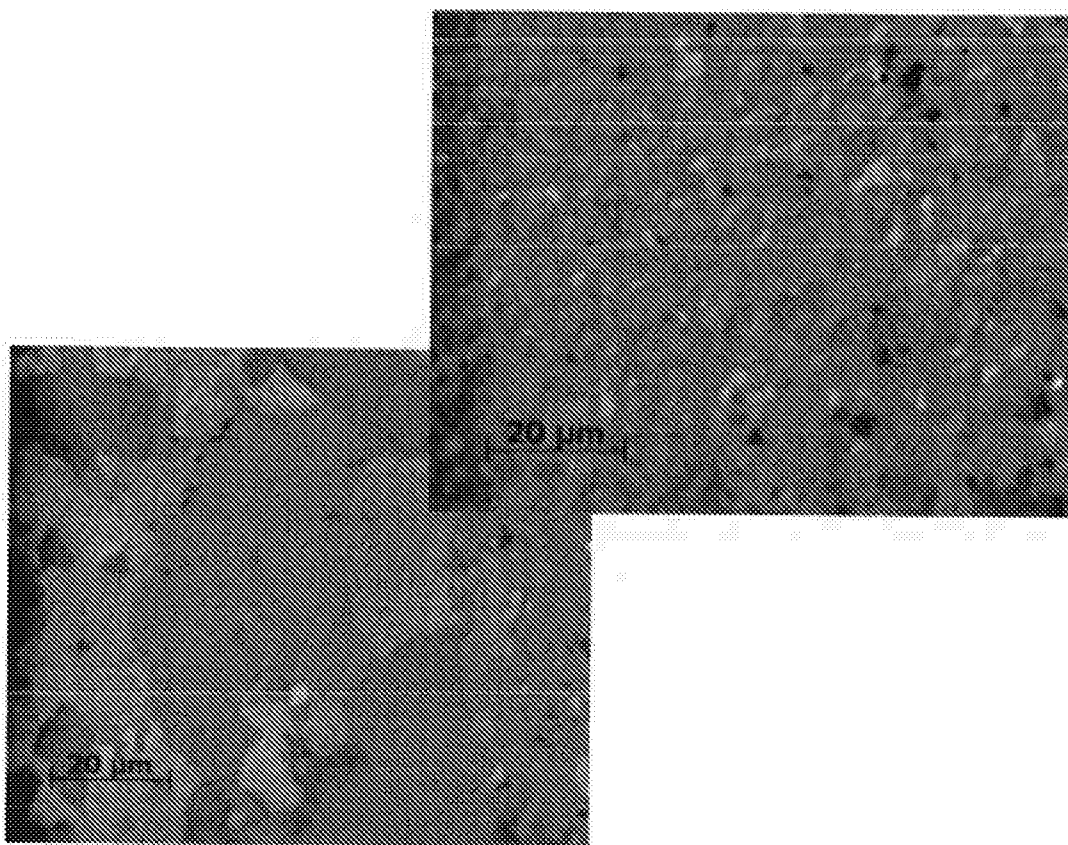


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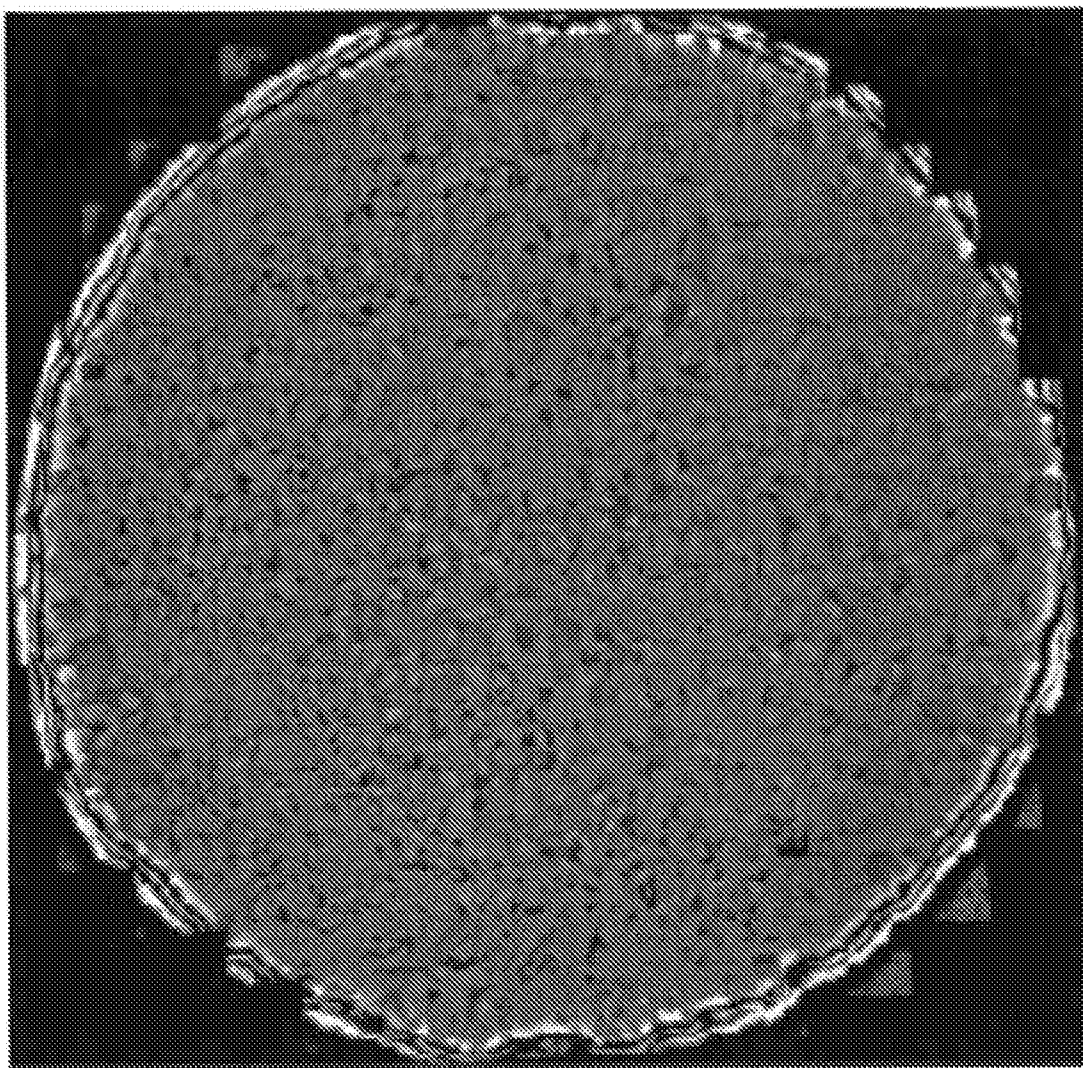


Figure 36

INTERNATIONAL SEARCH REPORT

International application No

PCT/GB2015/053704

A. CLASSIFICATION OF SUBJECT MATTER
 INV. B22F3/20 C22C1/04
 ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
 B22F C22C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2014/000332 A1 (WILSON ROBERT SIMON [AU] ET AL) 2 January 2014 (2014-01-02) paragraphs [0001] - [0007], [0013] paragraphs [0074], [0075], [0093] - [0095] -----	1,2,4-7, 9,11-21
X	US 4 468 945 A (FAIREY NORMAN R [GB]) 4 September 1984 (1984-09-04)	1,2,4-7, 9,10, 13-16, 19-21,25 26-28
Y	example 1 figures 1-4 column col. 2, lines 20-43 -----	
X	GB 2 329 602 A (T & N TECHNOLOGY LTD [GB]) 31 March 1999 (1999-03-31) page 2, paragraph 8 - page 4, paragraph 1 figure 1 ----- -/-	1,2,4-9, 17-24



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Date of the actual completion of the international search

26 February 2016

Date of mailing of the international search report

04/03/2016

Name and mailing address of the ISA/

European Patent Office, P.B. 5818 Patentlaan 2
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 Fax: (+31-70) 340-3016

Authorized officer

Forestier, Gilles

INTERNATIONAL SEARCH REPORT

International application No

PCT/GB2015/053704

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	Claudia Stadelmann: "Extrusion von Metallpulvern durch kontinuierliches Pulverstrangpressen", 19 November 2009 (2009-11-19), pages 1-145, XP055077332, Retrieved from the Internet: URL:http://www.opus.ub.uni-erlangen.de/opus/volltexte/2009/1466/pdf/Claudia_Stadelmann_Dissertation.pdf [retrieved on 2013-09-02] page 14 - page 16 page 22, paragraph 3.1 page 31, paragraph 3.3.2.1 - page 33 figures 2.5, 2.6 page 2; figure 1.1	1-7,9, 11-13, 19-21
X	----- Tomás Kubina ET AL: "PREPARATION AND THERMAL STABILITY OF ULTRA FINE-GRAINED COMMERCIALLY PURE TITANIUM WIRE", 1 January 2012 (2012-01-01), XP055253579, Retrieved from the Internet: URL:http://www.comat.cz/files/proceedings/11/reports/1301.pdf [retrieved on 2016-02-26]	29,30
Y	3.1 experimental processing 3.3 Annealing experiment figure 5	26-28
Y	----- GB 2 175 832 A (METAL BOX PLC) 10 December 1986 (1986-12-10) page 1, lines 5-30 page 4, lines 27-47 figure 1 -----	26-28

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/GB2015/053704

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 2014000332	A1	02-01-2014	AU 2012225201 A1 02-05-2013
		CA 2829391 A1 13-09-2012	
		CN 103415359 A 27-11-2013	
		EA 201391306 A1 30-05-2014	
		EP 2683502 A1 15-01-2014	
		JP 5818918 B2 18-11-2015	
		JP 2014507286 A 27-03-2014	
		KR 20140044314 A 14-04-2014	
		NZ 615063 A 25-07-2014	
		US 2014000332 A1 02-01-2014	
		WO 2012119196 A1 13-09-2012	

US 4468945	A	04-09-1984	AR 231807 A1 29-03-1985
		AU 548964 B2 09-01-1986	
		AU 8621282 A 27-01-1983	
		BR 8207752 A 31-05-1983	
		CA 1195289 A 15-10-1985	
		DD 202635 A5 28-09-1983	
		DE 3261955 D1 28-02-1985	
		DK 26383 A 03-02-1983	
		EP 0073101 A1 02-03-1983	
		ES 8400265 A1 16-01-1984	
		HK 26685 A 12-04-1985	
		IE 53043 B1 11-05-1988	
		IL 66356 A 30-08-1985	
		IN 158974 B 28-02-1987	
		JP S5825812 A 16-02-1983	
		KR 840000292 A 18-02-1984	
		MW 2982 A1 14-12-1983	
		MY 109985 A 31-12-1985	
		NZ 201362 A 31-07-1985	
		OA 7370 A 31-08-1984	
		PH 19790 A 02-07-1986	
		PT 75296 A 01-08-1982	
		SG 3485 G 14-06-1985	
		US 4468945 A 04-09-1984	
		WO 8300299 A1 03-02-1983	
		ZA 8205151 A 25-05-1983	
		ZW 14682 A1 12-01-1983	

GB 2329602	A	31-03-1999	AU 9179498 A 23-04-1999
		GB 2329602 A 31-03-1999	
		WO 9916560 A1 08-04-1999	
		ZA 9808869 A 30-03-1999	

GB 2175832	A	10-12-1986	GB 2175832 A 10-12-1986
		JP H021203 B2 10-01-1990	
		JP H0115325 B2 16-03-1989	
		JP S6317004 B2 12-04-1988	
		JP S6361091 B2 28-11-1988	
		JP S6361092 B2 28-11-1988	
		JP S59144518 A 18-08-1984	
		JP S59144519 A 18-08-1984	
		JP S59144520 A 18-08-1984	
		JP S59145704 A 21-08-1984	
		JP S62168610 A 24-07-1987	
		KE 3779 A 27-11-1987	
		SG 79287 G 15-04-1988	

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/GB2015/053704

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
		ZA 8400473 A	28-11-1984
		ZA 8400474 A	28-11-1984
