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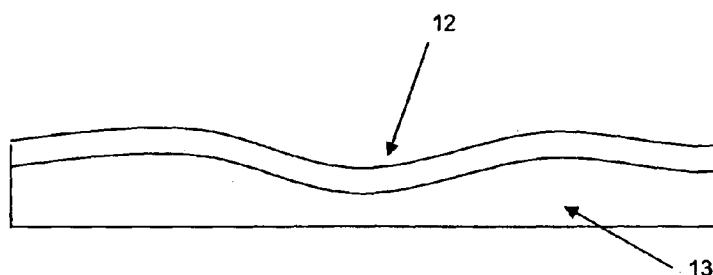


Figure 1

(57) Abstract: A composite lay up tool comprising a metal faceplate (12) on a support member (13), wherein the faceplate has been formed by cold-gas dynamic spraying of particles of the metal onto a substrate and wherein the support member is compatible with the faceplate and comprises a casting of a ceramic composition. A process for forming the tool is disclosed. In one aspect, a castable ceramic composition comprises a binder and an aggregate component mixable with water to form a slurry ready for casting, wherein the aggregate component is one of, or a mixture of, fused silica and a particulate metal alloy, and the binder and aggregate component are present in the proportions 10-50 wt% and 90-50 wt% based on the dry weight of the composition. Also disclosed is a process for producing a faceplate for a lay up tool, which process comprises cold-gas dynamic spraying of particles of a nickel-iron alloy onto a substrate to form a faceplate of the nickel-iron alloy on the substrate, the faceplate being of a thickness in the range 2 to 10 mm. Further disclosed is a process for depositing a metal on a faceplate of a lay up tool in which the faceplate is formed of a nickel-iron alloy, which process comprises preparing an area of a surface of the faceplate to effect cleaning and conditioning of the area to accept and adhere to cold-gas dynamic sprayed particles, and cold-gas dynamic spraying particles of the metal onto the cleaned and conditioned area, wherein the metal is a nickel-iron alloy selected to be compatible with the faceplate.



Formation, repair and modification of lay up tools

Field of the invention

The present invention relates to lay up tools useful in the manufacture of composite materials, to processes for making such tools and/or faceplates for such tools, and to
5 castable compositions useful in the making of such tools. In a particular aspect, the invention relates to the repair and/or modification of the faceplates of lay up tools. The invention is believed to have particular utility in relation to lay up tools employed to produce composite components for use in the aerospace and automobile industries.

Background of the invention

10 Fibre-based composite components, for example for use in the aerospace industry, are typically made by moulding a fibre-reinforced composite material on a lay up tool followed by curing of the material on the tool at elevated temperature (normally up to about 200°C) and under vacuum. After curing, the composite material is allowed to cool and then removed from the tool, and the tool is prepared for re-use. During this process,
15 the thermal expansion of the tool must be minimised to ensure minimal deformation of the composite component being produced.

The surface of the lay up tool that contacts the composite material is called a faceplate (or working surface or tooling surface) and this is required to be highly precise and designed to close tolerances. As manufacture of composite components on the lay up
20 tool involves high temperature curing, the working surface must also exhibit minimal dimensional change if high precision composite structures are to be produced repeatedly. The working surface must therefore be formed of a material having a low coefficient of thermal expansion. By way of example, the faceplate may be formed of specially selected nickel alloys or iron-based nickel-containing alloys. Invar 36 is one
25 alloy that is commonly used. Invar 36 is a 36% nickel - 64% iron alloy having a rate of thermal expansion approximately one-tenth that of carbon steel at temperatures up to 400°F (204°C).

Typically, the current process for the production of a faceplate involves machining a

monolithic block of a suitable material (such as Invar 36) to provide a faceplate having the shape and size required for the composite part being produced. However, this approach is very wasteful as material is reduced to swarf during the machining process. Additionally, once formed, the faceplate must then be secured (typically by welding) to a rigid structure (often termed an "egg crate") that provides support to the faceplate. This process can be somewhat involved and labour intensive. For compatibility reasons, the support is typically also made of the same material as the faceplate.

Lay up tools can be sizeable, especially in the aerospace industry where it is desired to produce large composite structures, and in this context the tools are particularly expensive to manufacture. The cost is mitigated somewhat by the fact that the tools are re-useable, but it would be desirable to minimise usage and wastage of expensive materials such as Invar 36.

Against this background it would be desirable to provide an alternative form of lay up tool that may be produced with relative ease, that reduces consumption of expensive materials and that, at least in one or more embodiments, provides at least the same level of performance when manufacturing composite components as tools produced by the conventional process described above.

Thermal cycling experienced by a lay up tool over its lifetime can lead to crack formation in the faceplate of the tool. Engineers sometimes also require modification of the original tool design in order to vary the shape of composite component being produced. This may be achieved by addition of material to the faceplate of the tool,

Currently, with lay up tools used in the manufacture of composite components, faceplate repair and modification is carried out by a welding process in which a region of the faceplate and material to be applied to it are melted and fused together using an arc-based welding system or laser. This approach is not ideal however because a heat affected zone is formed at the weld/faceplate interface. This zone does not have the same microstructure and properties as the faceplate and in turn this can cause dimensional instability when the tool is used repeatedly because of the thermal cycling that the tool is subjected to.

There is therefore the need to provide an alternative process for the repair and modification of a faceplate of a lay up tool for the manufacture of fibre-reinforced composites.

Summary of the invention

- 5 Accordingly, in a first aspect, the present invention provides a composite lay up tool comprising a metal faceplate on a support member, wherein the metal faceplate has been formed by cold-gas dynamic spraying of particles of the metal and wherein the support member is compatible with the faceplate and comprises a casting of a ceramic composition.
- 10 In a second aspect, the present invention provides a process for producing a composite lay up tool that comprises a metal faceplate on a support member compatible with the faceplate, which process comprises forming the metal faceplate by cold-gas dynamic spraying of particles of the metal onto a substrate and forming the support member by casting of a ceramic composition.
- 15 The present invention also provides, in a third aspect, a castable ceramic composition comprising a binder and an aggregate component mixable with water to form a slurry ready for casting, wherein the aggregate component is one of, or a mixture of, fused silica and a particulate metal alloy, and the binder and aggregate component are present in the proportions 10-50 wt% and 90-50 wt% based on the dry weight of the
- 20 composition.

The present invention also provides the use of a composite lay up tool in accordance with the invention in the manufacture of a composite component.

- The invention further provides, in a further aspect, a process for producing a faceplate for a lay up of tool, which process comprises cold-gas dynamic spraying of particles of a
- 25 nickel-iron alloy onto a substrate to form a faceplate of the nickel-iron alloy on the substrate, the faceplate being of a thickness in the range 2 to 10 mm.

In a fifth aspect, the invention still further provides a process for depositing a metal on a

faceplate of a lay up tool in which the faceplate is formed of a nickel-iron alloy, which process comprises preparing an area of a surface of the faceplate to effect cleaning and conditioning of the area to accept and adhere to cold-gas dynamic sprayed particles and cold-gas dynamic spraying particles of the metal onto the cleaned and conditioned
5 area, wherein the metal is a nickel-iron alloy selected to be compatible with the faceplate.

Herein the term "metal" is intended to embrace metals per se and metal alloys. Typically, however, the invention will be implemented using a metal alloy for forming, repairing or modifying the faceplate.

10 Throughout this specification and the claims which follow, unless the context requires otherwise, the word "comprise", and variations such as "comprises" and "comprising", will be understood to imply the inclusion of a stated integer or step or group of integers or steps but not the exclusion of any other integer or step or group of integers or steps.

The reference in this specification to any prior publication (or information derived from
15 it), or to any matter which is known, is not, and should not be taken as an acknowledgment or admission or any form of suggestion that that prior publication (or information derived from it) or known matter forms part of the common general knowledge in the field of endeavour to which this specification relates.

Brief description of the drawings

20 Embodiments of the present invention are illustrated with reference to the accompanying non-limiting drawings in which:

Figure 1 is a schematic side elevation of a lay up tool according to an embodiment of the first aspect of the invention;

Figure 2 is a schematic showing a modular support member (egg-crate) for another
25 embodiment of lay up tool according to the first aspect of the invention; and

Figures 3 to 6 are schematics illustrating successive steps in the formation of a composite lay up tool in accordance with a further embodiment of the present invention;

Figure 7 is an optical image of a cross-sectioned Invar 36 faceplate repaired according to an embodiment of the fifth aspect of the invention;

5 Figure 8 is a diagram showing a surface profile of an Invar 36 faceplate that has been prepared for cold-gas dynamic spraying in accordance with an embodiment of the fifth aspect of the present invention; and

Figure 9 is a graph showing the relationship between the adhesive strength of Invar 36 cold sprayed onto an Invar 36 faceplate and the pitch of the surface profile of the
10 substrate formed according to Figure 8 prior to cold spraying.

Detailed description of the embodiments of the invention

Figure 1 illustrates an embodiment of lay up tool according to the first aspect of the invention, comprising a profiled metal faceplate 12 on a support member 13. The faceplate is composed of particles of the metal assembled by cold-gas dynamic
15 spraying, while the support member 13 is a casting of a ceramic composition.

As mentioned, faceplate 12 is formed by cold-gas dynamic spraying of metal particles. As will be explained in more detail below, the nature of the support member 13 may vary depending upon the order in which the individual components of the composite lay up tool are produced.

20 Cold-gas dynamic spraying (also referred to herein as cold-gas spraying or simply as cold spraying) is a known process for applying coatings to surfaces. In general terms the process involves feeding (metallic and/or non-metallic) particles into a high pressure (typically > 30 bar), gas flow stream which is then passed through a converging/diverging nozzle that causes the gas stream to be accelerated to supersonic
25 velocities. The particles are then directed on to a surface to be coated. The process is carried out at relatively low temperatures, below the melting point of the particles and the substrate to be coated, with a coating being formed as a result of particle

impingement on the substrate surface. The fact that the process takes place at relatively low temperature allows thermodynamic, thermal and/or chemical effects on the surface being coated and on the particles making up the coating to be reduced or avoided. This means that the original structure and properties of the particles can be
5 preserved without phase transformations etc that might otherwise be associated with high temperature coating processes such as plasma, HVOF, arc, gas-flame spraying or other thermal spraying processes. The underlying principles, apparatus and methodology of cold-gas dynamic spraying are described, for example, in US patent 5,302,414.

- 10 The cold-gas dynamic spraying process by which the faceplate is formed may be automated and offers great flexibility with respect to implementation especially when large tools are involved.

The metal employed to form the faceplate of the lay up tool is preferably selected for its suitability in the production of composite components. One important property that
15 contributes to such suitability is low thermal expansion coefficient, for example in the range 0.6-1.9 $\mu\text{m}/^\circ\text{C}$ up to 200°C.

The faceplate will typically be formed of a metal alloy, such as a nickel alloy or an iron-based nickel containing alloy. Alloys that are suitable for faceplates are well known in the art. By way of example, the metal alloy may be a nickel-iron alloy containing from
20 30-50wt% nickel, such as from 36-42wt% nickel. Invar 36 (Ni 36 wt%, Fe 64 wt%) is a preferred material to use.

Usually, the particle size of the metal alloy cold sprayed will range from 5 to 50 μm , for example from 8 to 30 μm , with a D_{50} particle size of about 20 μm . Particles useful in the process of the invention are commercially available (for example from ESPI Metals,
25 Ashland, Oregon USA 97520). The sprayed particles may be spherical, non-spherical regular shaped or irregularly shaped, but spherical shape is preferred.

The faceplate thickness is typically 2-10 mm.

The operating parameters for the cold spraying process may be manipulated in order to

achieve a faceplate having desirable properties. Operating parameters that may be varied include temperature, pressure, stand-off (distance between cold spray nozzle and faceplate surface), and spray angle. The particle size and morphology may also be varied. Upon impact of particles on a substrate during cold spraying the particles
5 plastically deform and bond to the substrate and to each other. The amount of particle deformation will vary depending on the velocity of impact, particle size and shape, substrate and particle materials.

The apparatus used for cold spraying is likely to be of conventional form, and such equipment is commercially available. In general terms the basis of the apparatus used
10 for cold spraying will be as described and illustrated in US patent 5,302,414. It may of course be necessary to modify the apparatus, for example, when it is desired to apply the cold spraying process to a large workpiece. However, the process principles remain unchanged.

The support member 14 is cast from a ceramic composition. In the composite lay up
15 tool of the invention the support member and faceplate are preferably in contact with each other or in very close proximity to each other, and it is highly preferred that, in at least a selected temperature range (e.g. 150-250°C) the support member has the same, or essentially the same, i.e. similar, ($\pm 15\%$), thermal expansion coefficient as the metal in which the faceplate is formed. Matching of the thermal expansion coefficient of the
20 support member to the faceplate is intended to minimise dimensional instability and associated stresses in the composite tool when the tool is subjected to thermal cycling as occurs during use. It is highly preferred that the lay up tool retains its original dimensions and tolerances to ensure consistency in the composite components produced by use of the tool.

25 In an embodiment of the invention the ceramic composition comprises a suitable binder, such as cement, e.g. Portland cement, and an aggregate component that enables matching of the thermal expansion coefficient of the (cast) support member to the faceplate. The ceramic composition should also provide a support member that is sufficiently strong and resilient to provide stable support to the faceplate and composite
30 material being moulded on the lay up tool. In an embodiment of the invention the

aggregate may be fused silica. In another embodiment the aggregate may be a metal e.g. metal alloy, that is the same as or similar to (in terms of relevant properties) to the metal used for the faceplate. When the aggregate is a metal alloy, this may take the form of what would otherwise be waste material, for example swarf. In an embodiment
5 of the invention the aggregate component comprises a nickel iron alloy such as Invar 36, which is well suited to formation of a faceplate of the same metal alloy.

As a general guide, the amount of binder (e.g. cement) is typically 10 to 50 wt% and the amount of aggregate is 50-90% based on the dry weight of the composition. The binder and aggregate component may be mixed with water to form a castable slurry. The
10 amount of water used is typically 5-20 wt%. After mixing with water the composition is in a form ready to be cast, after which the composition is allowed to set/cure. The time taken for the composition to set will vary but is typically a number of days, for example 5-7 days. Setting involves an exothermic reaction and the extent of setting can be followed by monitoring the temperature of the composition. After setting has been
15 completed, the cast composition can then be subjected to hot air drying. This is usually carried out at a temperature of up to about 100°C. This step can take a number of hours and is usually undertaken until no further weight change exists at 100°C. It may also be necessary to subject the cast composition to a final sinter at a temperature of up to about 250°C over a period of a few hours to drive off all traces of moisture.

20 It should be noted that air bubbles in the cast composition can act as stress concentration points and it is therefore desirable to minimise the amount of air that will be trapped in the cast composition. This can be done by formulating the composition for casting under vacuum, pouring the composition slowly when casting and/or vibrating the composition immediately after casting (by vibration of the mould in which the
25 composition is cast).

It should also be noted that the cast composition may be subject to hygroscopic degradation and it is important therefore to carry out regular checks on the integrity of the cast composition.

The composite lay up tool may take a variety of forms each involving a different

sequence of formation steps. In one embodiment the support member is produced using a suitably shaped mould. Once produced the support member can be shaped (by machining or water jet cutting) to produce a desired surface profile. A faceplate may then be developed by cold spraying of metal particles onto the shaped surface of the support member. Figure 1 illustrates an exemplary lay up tool thereby produced. Typically, the dimensions of the faceplate as-produced will exceed specification and the faceplate is then finished by machining to achieve the desired dimensions and tolerances.

In a related embodiment the support member may be a modular structure made up of a number of pre-formed units. Each unit is formed by casting from a ceramic composition as described, with the units then being joined together using fasteners and the like to form a modular structure. This approach may provide more flexibility in terms of manufacturing and design of the support member. An exemplary modular support member is shown schematically in Figure 2.

In another embodiment for producing a lay up tool as depicted in Figure 1, the faceplate is formed first by cold spraying of metal alloy particles on to a suitably shaped pre-formed mould. The mould defines the intended surface profile of the faceplate and is typically made of aluminium or steel. After production of the faceplate, the support member is formed by casting of ceramic composition on to the faceplate. For dimensional stability it may be desirable to maintain the faceplate in contact with the mould used for its formation during casting and subsequent steps in forming the support member. A surrounding mould having suitable wall members will be needed during casting of the ceramic composition. After the support member has been formed, the faceplate is then separated from the mould. This may be done by machining of the mould away from the faceplate, or it may be possible to chemically dissolve the mould material. For example, an aluminium mould may be dissolved using sodium hydroxide. Of course, care must be taken during this step to ensure that the faceplate is not compromised. Surface finishing of the faceplate (by machining) is generally undertaken.

In another embodiment, the support member (possibly modular in nature) and faceplate

are each produced independently and then joined together subsequently. In this case the support member may be formed by simple moulding and the faceplate formed by cold spraying of metal alloy particles onto a suitable mould or other substrate with the mould or substrate subsequently being removed.

- 5 In an embodiment of the invention bonding between the support member and faceplate may be enhanced by the use of anchors that are attached to the faceplate and that extend into the support member. These anchors may be produced by cold spraying on the surface of the faceplate or anchors may be attached to the faceplate by conventional means such as by screwing or welding. When the anchors are formed on
10 or attached to the faceplate, the ceramic composition will subsequently be cast over the anchors. This process is explained in more detail with reference to Figures 3 to 6.

Figure 2 shows a layer of metal alloy (2) (e.g. Invar 36) that has been applied to a mould (3) (e.g. aluminium or steel) by cold spraying, to form a faceplate. The mould (3) has a desired surface profile.

- 15 In the step depicted in Figure 4, a series of Y-shaped anchors (4) are attached (e.g. by cold spraying, screwing or spot welding) to the surface of the faceplate (2) remote from the mould. It will be appreciated that this surface is the underside of the faceplate (2) in the subsequent tool. The anchors (4) are typically formed of the same material as the faceplate to avoid any dimensional instability during thermal cycling of the lay up tool.
- 20 Figure 5 shows the next step in the process in which a suitably selected ceramic composition (5) is poured into a mould (6) and over the anchors (4). The ceramic composition (5) is then allowed to set and dried. The surrounding mould (6) is then taken away and the mould (3) used for forming the faceplate (2) removed, by machining or by chemical dissolution. The composite lay up tool as-produced is shown in Figure 6.
- 25 Surface finishing of the faceplate (2) will be undertaken before the tool is used for composite production.

In a related embodiment, the anchors may be embedded in the ceramic composition during casting with a portion of each anchor extending from the surface of the casting.

After suitable shaping of the surface of the casting, a faceplate can then be developed by cold spraying. The anchors will become embedded in the faceplate during cold spraying.

5 In another embodiment of the invention the composite lay up tool preferably has an air gap (G) (Figures 5 and 6) extending between the support member and faceplate. This air gap (G) may be beneficial in terms of buffering heat transfer between the faceplate and support member during manufacture of composite materials on the faceplate. This air gap (G) is usually of uniform dimension over the area of the tool, and may be up to 30 mm to allow space for vacuum lines and other services for the tool assembly

10 This air gap (G) may be formed by a modification of the production process illustrated in Figures 3 to 6. Thus, prior to casting of the ceramic composition (Figure 5) a layer of sacrificial material (typically a polymer such as polystyrene) is provided over the surface of the faceplate upon which the ceramic composition is to be cast. After casting, drying etc, to produce the support member, the sacrificial material can be removed (e.g. it can
15 be dissolved with suitable chemical or burned off at elevated temperature). This leaves an air gap (G) between the support member and faceplate, with the faceplate being supported by the anchors embedded in the support member. It may be possible to achieve the air gap (G) with modification to the other production processes described. Figures 5 and 6 show this air gap but it will be appreciated that the invention may be
20 implemented without the air gap and these figures should be understood accordingly.

It has been found that cold-gas spraying enables metal to be deposited on the surface of a nickel-iron faceplate, e.g. of Invar 36, by a solid-state deposition process in which the deposited metal can exhibit material properties that are the same as, or suitably the same as, the original faceplate. In this context the expression "suitably the same"
25 means that the deposit may not be identical to the original faceplate in microstructure and properties but minor differences may be tolerated provided they do not adversely impact performance of the lay up tool.

It is important to the repeated use of the faceplate that the deposited material retains the same, or suitably the same, chemistry and thermal expansion coefficient of the

material from which the faceplate was originally formed. Moreover, it has been found that the metal deposited by cold spray is of low residual stress and presents no undercut issues at the deposit/substrate interface which would otherwise have potential to cause cracking. As before, the cold-gas spraying process may be automated and offers great flexibility with respect to implementation especially when large tools are involved. The faceplate to be treated by the repair or modification process of the invention does not require pre-heating before cold spraying and the low temperature regime involved avoids oxide formation in the deposited metal. The result is improved product quality.

10 In one embodiment the modification of the faceplate involves repairing a defect in a faceplate. The defect may be a crack produced by thermal cycling during use of the faceplate, a worn/eroded area, or an indentation or other form of mechanical damage on the faceplate surface. In accordance with this embodiment the present invention provides a process for repairing a defect on a nickel-iron faceplate of a lay up tool, which process comprises cold gas dynamic spraying of particles of a compatible nickel-iron alloy over the defect. According to this embodiment, a defect on a faceplate can be remediated so that original surface design and finish can be achieved.

In this embodiment the preferred approach is to deposit metal over the defect and a small area around the defect. This may assist the deposited material to form a strong bond to the faceplate. Prior to cold spraying the area to be treated will usually be prepared by machining, grit blasting, water jet blasting and the like, so that the area is suitably clean and conditioned to receive cold sprayed particles. If the area to be treated is not suitably clean or includes loose matter or debris, adhesion of cold spray particles may be impaired. If the defect is a crack or similar, the faceplate is machined to form a recess that eliminates and replaces the crack.

Usually, the repair involves cold spraying of metal such that the original dimensions of the area being repaired are exceeded. Subsequently, the area can be machined to restore original dimensions and tolerances, for example by milling or grinding.

Figure 7 shows an Invar 36 faceplate (21) that has been repaired using cold spray. A pit

on the surface of the faceplate has been filled (22) by cold spraying of metal alloy particles. There is an interface (23) between the original faceplate material (21) and the deposited metal (22) but this does not create dimensional instability since there no localised heating at the interface that will lead to formation of a heat affected zone or distortion of the tool. There are no undercuts at the substrate/deposit interface (usually associated with weld repairs). Cold spray deposits are also vacuum tight which is an important property for composite part manufacture. It will be seen in Figure 7 that the prepared area in a recess defined by surfaces that later form the aforesaid interface (23). These interfaces include a flat broad conical surface (25) inclined at no greater than 45° to the adjacent unprepared faceplate surface (26), and a curved or concave bottom (27) of a radius similar to or larger than the depth of the recess. This facilitates successful depositing and adhesion of the sprayed particles.

In another embodiment of this aspect of the invention cold spraying may be used to add a (new) structure to, or modify an existing structure of, a faceplate in order to change its overall surface design. This may be necessary when it is desired to produce a composite of different design without needing to incur the cost of producing a new faceplate. In this embodiment the present invention provides a process for modifying (or re-designing) a faceplate of a lay up tool, which process comprises depositing a compatible metal onto the faceplate in a desired configuration.

Prior to cold spraying the area of the faceplate to be modified will normally be prepared, for example as described above. Usually, the deposited (structure) produced by cold spraying will have width dimensions in excess of what is actually required. Machining (e.g. milling or grinding) may then be carried out to obtain the desired dimensions and tolerances.

The metal alloy that is deposited by cold spraying is referred to as being a "compatible metal alloy". What this means is that once deposited on a faceplate the metal alloy behaves the same as, or suitably the same as, the metal alloy of the faceplate with respect to those key characteristics that impact on performance of the faceplate as a lay up tool. One very important characteristic is thermal expansion coefficient. Here the metal alloy for cold spraying has the same, or essentially the same ($\pm 5\%$), thermal

expansion coefficient as the metal alloy from which the faceplate is formed. Preferably, the metal alloy that is cold sprayed is the same metal alloy used for the faceplate. Thus, if the faceplate is made of Invar 36, the metal alloy for cold spraying is also Invar 36.

- 5 As with earlier described aspects of the invention, the particle size of the metal alloy to be cold sprayed will range from 5 to 50 μm , for example from 8 to 30 μm , with a D_{50} particle size of about 20 μm . Particles useful in the process of the invention are commercially available (for example ESPI Metals, Ashland, Oregon USA 97520). The sprayed particles may be spherical, non-spherical regular shaped or irregularly shaped
10 but spherical shape is preferred.

In an embodiment of the invention a surface preparation technique has been developed that achieves enhanced adhesion of cold sprayed particles to a faceplate formed in a nickel-iron alloy. The technique involves preparing the area to be cold sprayed so that it has a repeating pattern of grooves (or channels). Noting the particle size referred to
15 above for the particles to be deposited by cold spraying, the grooves preferably have an aspect ratio (pitch:depth) in the range 4:1 to 9:1. The grooves may be formed by machining and may have a variety of cross-sectional shapes (e.g. V-shaped, U-shaped or rectangular). As a general guide the pitch is typically 0.1 to 1.0 mm, preferably from 0.3 to 0.9 mm. The presence of the grooves is believed to increase interlocking of cold
20 sprayed particles with the substrate thereby increasing the overall bond strength of the surface modification.

Figure 8 is a schematic showing a surface prepared for cold spraying in accordance with this embodiment. The pitch (S_m) is measured as a repeat unit over the surface. The groove depth is denoted R_z .

- 25 It is highly preferred that the particles that are cold sprayed adhere strongly to the faceplate and that the deposited material is dense and defect-free. The operating parameters for the cold spray process may be manipulated in order to achieve a deposit having desirable properties. Operating parameters that may be varied include temperature, pressure, stand-off (distance between cold spray nozzle and faceplate

surface), and spray angle. The particle size and morphology may also be varied. Upon impact of particles on the faceplate the particles plastically deform and bond to the surface of the faceplate and to each other. The amount of particle deformation will vary depending on the velocity of impact, particle size and shape, substrate and particle materials.

As with earlier described aspects of the invention, apparatus used for cold spraying is likely to be of conventional form, and such equipment is commercially available. In general terms the basis of the apparatus used for cold spraying will be as described and illustrated in US 5,302,414. It may of course be necessary modify the apparatus, for example, when it is desired to apply the cold spray process to a large work piece. However, the process principles remain unchanged.

Embodiments of the present invention are illustrated with reference to the following non-limiting examples.

Example 1 – Properties of materials

Details of the raw materials used for the experimental work are shown in Table 1

Table 1 Details of materials Used for CTE measurements

Materials		Supplier	Specification
Wrought Invar 36 Specimen		Commercial available Invar block 36	Fe: 64% Ni: 36%
cold sprayed Invar 36 specimen		ESPI metals	Fe: 64% Ni: 36%
Portland cement		Australian Builders	Ecoblend, type GB
Invar machining swarf		Swarf obtained from client after machining wrought Invar 36	0.425-4 mm
Fused silica	granules	Linyi Agrishine Import & Export Co. Ltd.	5 -10 mm Dia.
	sand		0 – 1 mm Dia

5 1. Compositions

The compositions of the composite specimens are shown in **Table 2**.

Table 2 Compositions of Specimens Prepared for CTE measurements

Raw materials	Wt %	
	Invar swarf composite	Fused silica composite
Cement	32.45	14.29
Invar swarf	67.55	0
Fused silica lumps	0	57.14
Fused silica sand	0	28.57
Total	100	100
Water (parts)	10	8.9

Method for preparation of CTE test samples for Invar 36 swarf and fused silica composites

- 5
 - The solid parts of the composite were dry mixed in a beaker using a spatula.
 - Water was added gradually and thoroughly mixed.
 - The slurry was poured into a steel mould and vibrated for 5 minutes.
 - The mould was placed in a plastic bag and sealed to retain the moisture during curing
- 10
 - After minimum of 6 days of curing at room temperature, the block was removed from the mould
 - Test specimens 10×10× 25mm were machined from the dried composite block.

2. Thermal expansion coefficient (TEC) measurements

Test specimens were dried at 100°C overnight in an oven before commencing CTE measurements. Details of specimens are shown in Table 3, and test results in Table 4.

Table 3 Details of the specimens for TEC measurement

Specimen	Specimen size (mm)	density	Remarks
Wrought Invar	10 × 11 × 25	7.89 (measured)	
Cold sprayed Invar	10 × 10 × 25	≈ 8.1	
Invar swarf composite	10 × 10 × 25	3.65	
Fused silica composite	11.1×11.3×25.7	2.0 – 2.2	Density of the fused silica: 2.2

The CSIRO Dialtronic 6548 (Theta industries Inc) was used to measure the thermal expansion coefficient CTE of the specimens. Sample sizes were 10 high × 10 wide × 5 25 mm long. The Temperature range was from room temperature to 250°C. The heating rate was 5°C/min, and the atmosphere air.

The results are set out in Table 4.

Table 4 TEC Measurements

Material	Thermal expansion coefficient (CTE)			
	(µm/m-°C)			
	100°C	150°C	200°C	
*Commercial wrought Invar 36	1	1.2	1.8	
*Cold sprayed Invar	0.3	0.7	1.6	
*Composite Invar Swarf	0.3	0.5	0.9	
*Composite Fused Silica	1.9	1.4	0.6	

Example 2 – Manufacture of Aerospace Tooling Materials

Castable ceramic compositions which have a similar or lower thermal expansion coefficient as that of Invar 36 have been developed. The ceramic composition comprises cement with Invar 36 swarf and /or fused silica as aggregate(s). The thermal expansion coefficient of the cast composition (cement + Invar 36 aggregate, cement + fused silica aggregate and cement + Invar 36 aggregate + fused silica aggregate) were found to match the thermal expansion coefficient of wrought cast Invar 36.

The ceramic composition was formed by mixing cement with either Invar 36 swarf and/or fused silica aggregate. The mixing ratio of cement was 10-50 wt% with 50-90 wt% Invar 36 aggregate. Other materials can be added to the Invar 36 composite mixture or alternatively materials such as fused silica (5-20 wt% based on the dry weight of components) can replace a portion of the Invar 36 material to achieve tools with low coefficient of thermal expansion.

Prior to wet mixing the components were dry-mixed together using a tumbler machine for 0.5 hours followed by water addition of 5-20 wt%. The slurry mixture was then poured into a tool mould having the desired shape and vibrated for 5-30 minutes to remove any air bubbles (porosity) formed in the slurry. The slurry material in the mould was cured at room temperature for a minimum of 6 days. After curing the material was removed from the mould to reveal a monolithic piece. To achieve the final support member properties further drying was carried out overnight in an oven at 100°C followed by firing up to 250°C for 1 hour. The faceplate surface can then be produced on the surface of the support member by cold spraying of particles of Invar 36, or by surface coating with other coating materials prior to the production of carbon composite parts as described in Example 3.

Example 3 – Manufacture of Face-plate Tooling

Invar 36 deposits were obtained using a commercial CGT Kinetics 4000 series Cold spray system with a tungsten carbide MOC De Laval nozzle and nitrogen as propellant gas. The gun was equipped with a short pre-chamber tube. Gas pressures of 3.8 MPa with gas temperatures 800°C were used. The standoff distance was kept constant at 30 mm while the traverse speed was 1 m/s with overlapping passes of 2.0 mm. Following Cold spray application the Invar 36 deposit were machined to the same surface datum as the faceplate.

Suitably selected ceramic compositions were cast into moulds and shaped to provide a surface with desired profile. The surface was then sealed by cold spraying of particles of Invar 36. Other materials, including ferrous metals and in some cases non-ferrous metal powder (e.g. Ni, Al powder) may alternatively be used. The cold sprayed surface can be final machined or polished to the desired tolerance. It is necessary to finish all cold spray deposits by machining and/or polishing to maintain dimensional tolerance.

Example 4 - Repair or Modification of Invar 36 faceplate

To enhance the bond strength of cold sprayed Invar 36 deposits on existing Invar 36 tooling, a surface preparation technique has been developed which involves surface

profiling the substrate using conventional machining to form a particular pattern on the surface, as illustrated in Figure 8. The method increases the cold sprayed particle interlocking with the substrate thereby improving the overall bond strength of the repair or modification. By maintaining an average aspect ratio from 4 to 9 with respect to pitch and depth, the bond strength is proportional to the pitch used. Bond strengths increased more than 70% from approximately 20 MPa to approximately 34 MPa when the pitch profile was increased, (Figure 9).

Invar 36 deposits were obtained using a commercial CGT Kinetics 4000 series cold spray system with a tungsten carbide MOC De Laval nozzle and nitrogen as propellant gas. The gun was equipped with a short pre-chamber tube using gas pressures of 3.8 MPa with gas temperatures 800°C. The standoff distance was kept constant at 30 mm while the traverse speed was 1 m/s with overlapping passes of 2.0 mm. Following cold spray application to an Invar 36 faceplate, the cold sprayed Invar 36 deposit was machined to the same surface datum as the faceplate.

CLAIMS

1. A composite lay up tool comprising a metal faceplate on a support member, wherein the faceplate has been formed by cold-gas dynamic spraying of particles of the metal onto a substrate and wherein the support member is compatible with the
5 faceplate and comprises a casting of a ceramic composition.
2. A composite lay up tool according to claim 1 wherein the support member has a thermal expansion coefficient that differs from that of the faceplate by no greater than \pm 15% in at least a selected temperature range.
3. A composite lay up tool according to claim 2 wherein the selected temperature
10 range is 150-250°C.
4. A composite lay up tool according to claim 1, 2 or 3 wherein the ceramic composition comprises a binder and an aggregate component and the aggregate component is one of, or a mixture of, fused silica and a particulate metal alloy.
5. A composite lay up tool according to claim 4 wherein the binder and the
15 aggregate component are present in the proportions by weight 10-50% and 90-50% respectively, based on the dry weight of the composition.
6. A composite lay up tool according to any one of claims 1 to 5 wherein the metal of the faceplate is a nickel-iron alloy.
7. A composite lay up tool according to claim 6 wherein the metal of the faceplate is
20 Invar 36.
8. A composite lay up tool according to claim 6 or 7 wherein the ceramic composition includes an aggregate component comprising or particulate form of the same nickel-iron alloy.
9. A composite lay up tool according to any one of claims 1 to 8 wherein the
25 faceplate and the support member are connected by a plurality of anchors.

10. A composite lay up tool according to claim 9 wherein the faceplate and the support member are spaced apart by a predetermined gap.
11. A process for producing a composite lay up tool that comprises a metal faceplate on a support member compatible with the faceplate, which process comprises forming the metal faceplate by cold-gas dynamic spraying of particles of the metal onto a substrate and forming the support member by casting of a ceramic composition.
12. A process according to claim 11 wherein the metal and the ceramic composition are selected so that the support member has a thermal expansion coefficient that differs from that of the faceplate by no greater than $\pm 15\%$ in at least a selected temperature range.
13. A process according to claim 12 wherein the selected temperature range is 150-250°C.
14. A process according to claim 11, 12 or 13 wherein the ceramic composition comprises a binder and an aggregate component and the aggregate component is one of, or a mixture of, fused silica and a particulate metal alloy.
15. A process according to claim 14 wherein the binder and the aggregate component are present in the proportions by weight 10-50% and 90-50% respectively, based on the dry weight of the composition.
16. A process according to any one of claims 11 to 15 wherein the metal of the faceplate is a nickel-iron alloy.
17. A process according to claim 16 wherein the metal of the faceplate is Invar 36.
18. A process according to claim 16 or 17 wherein the ceramic composition includes an aggregate component comprising a particulate form of the same nickel-iron alloy.
19. A process according to any one of claims 11 to 18 including positioning a plurality of anchors to correct the faceplate and the support member.

20. A process according to any one of claims 11 to 19 wherein the substrate is the support member.
21. A process according to any one of claims 11 to 19 wherein the formed faceplate is mounted to the support member after its formation on the substrate, and the substrate is thereafter removed.
22. A castable ceramic composition comprising a binder and an aggregate component mixable with water to form a slurry ready for casting, wherein the aggregate component is one of, or a mixture of, fused silica and a particulate metal alloy, and the binder and aggregate component are present in the proportions 10-50 wt% and 90-50 wt% based on the dry weight of the composition.
23. A castable ceramic composition according to claim 22 where the particulate metal alloy is a nickel-iron alloy.
24. A castable ceramic composition according to claim 23 wherein the nickel-iron alloy is Invar 36.
25. A castable ceramic composition according to claim 22, 23 or 24 wherein the binder is a cement.
26. A castable ceramic composition according to any one of claims 22 to 25 wherein the composition when cast has a thermal expansion coefficient in the range 0.6 to 1.9 $\mu\text{m}/^\circ\text{C}$, at least over a selected temperature range.
27. A castable ceramic composition according to claim 26 wherein the selected temperature range is 0-200°C.
28. A ceramic support member cast in the castable ceramic composition of any one of claims 22 to 27.
29. The use of a composite lay up tool according to any one of claims 1 to 10 in the manufacture of a composite component.

30. A process for producing a faceplate for a lay up tool, which process comprises cold-gas dynamic spraying of particles of a nickel-iron alloy onto a substrate to form a faceplate of the nickel-iron alloy on the substrate, the faceplate being of a thickness in the range 2 to 10 mm.
- 5 31. A process according to claim 30 wherein the substrate is a compatible support member whereby the faceplate and the substrate comprise a lay up tool.
32. A process according to claim 30 or 31 wherein the nickel-iron alloy is Invar 36.
33. A composite lay up tool comprising a support member and a faceplate formed by the process of claim 30, 31 or 32.
- 10 34. A composite lay up tool comprising a support member and a faceplate that comprises an assembly of cold-gas sprayed particles of a nickel-iron alloy.
35. A composite lay up tool according to claim 33 or 34 wherein the nickel-iron alloy is Invar 36.
- 15 36. The use of a composite lay up tool according to any one of claims 33, 34 or 35 in the manufacture of a composite component.
37. A process for depositing a metal on a faceplate of a lay up tool in which the faceplate is formed of a nickel-iron alloy, which process comprises preparing an area of a surface of the faceplate to effect cleaning and conditioning of the area to accept and adhere to cold-gas dynamic sprayed particles, and cold-gas dynamic spraying particles
20 of the metal onto the cleaned and conditioned area, wherein the metal is a nickel-iron alloy selected to be compatible with the faceplate.
38. A process according to claim 37 wherein said preparation includes roughening said area of the faceplate surface.
- 25 39. A process according to claim 37 or 38 wherein said preparation includes forming a repeating pattern of grooves in said area.

40. A process according to claim 39 wherein the grooves have an aspect ratio (pitch:depth) in the range 4:1 to 9:1.
41. A process according to claim 39 or 40 wherein the pitch of the groove pattern is in the range 0.1 to 1.0 mm.
- 5 42. A process according to any one of claims 37 to 41 for repairing a crack or other defect in the faceplate, wherein said area is of dimensions that exceed the dimensions of the crack or other defect and wherein the process includes machining the deposited metal to restore original dimensions and surface of the faceplate.
- 10 43. A process according to any one of claims 37 to 42 wherein the prepared area itself is a recess defined by surfaces that are inclined no more than 45° to the adjacent unprepared faceplate surface and/or curved with a radius similar to or larger than the depth of the recess.
44. A composite lay up tool upon which a compatible material has been deposited by the process of any one of claims 37 to 43.
- 15 45. The use of a composite lay up tool according to claim 44 in the manufacture of a composite component.

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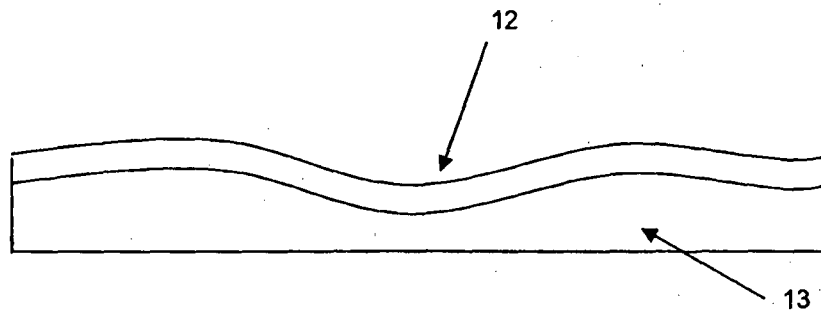


Figure 1

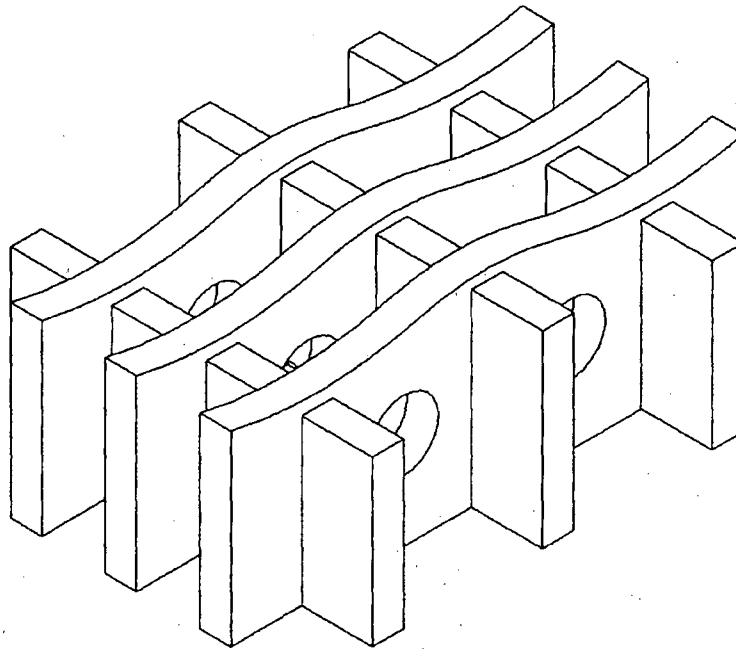


Figure 2

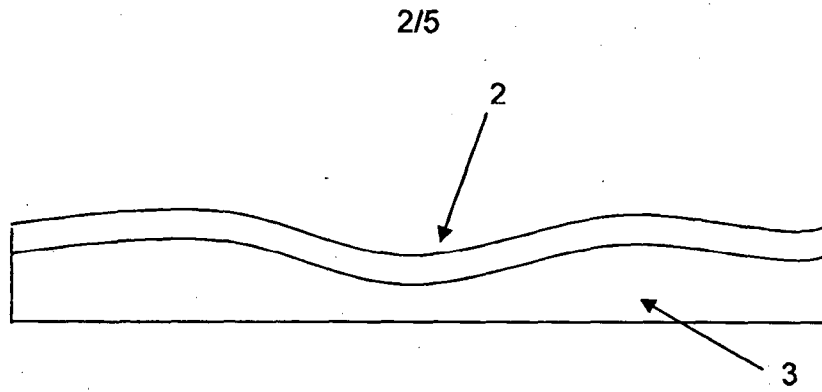


Figure 3

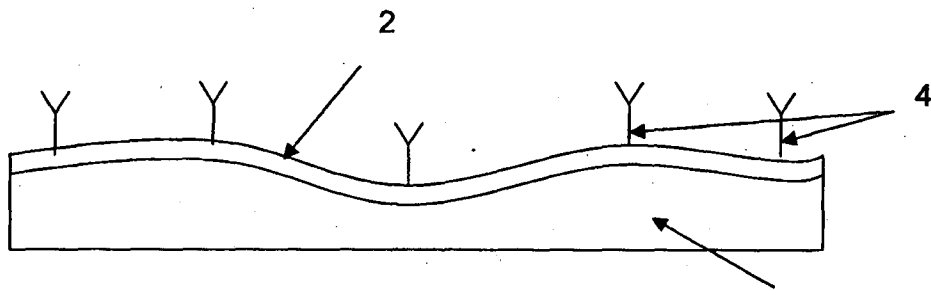


Figure 4

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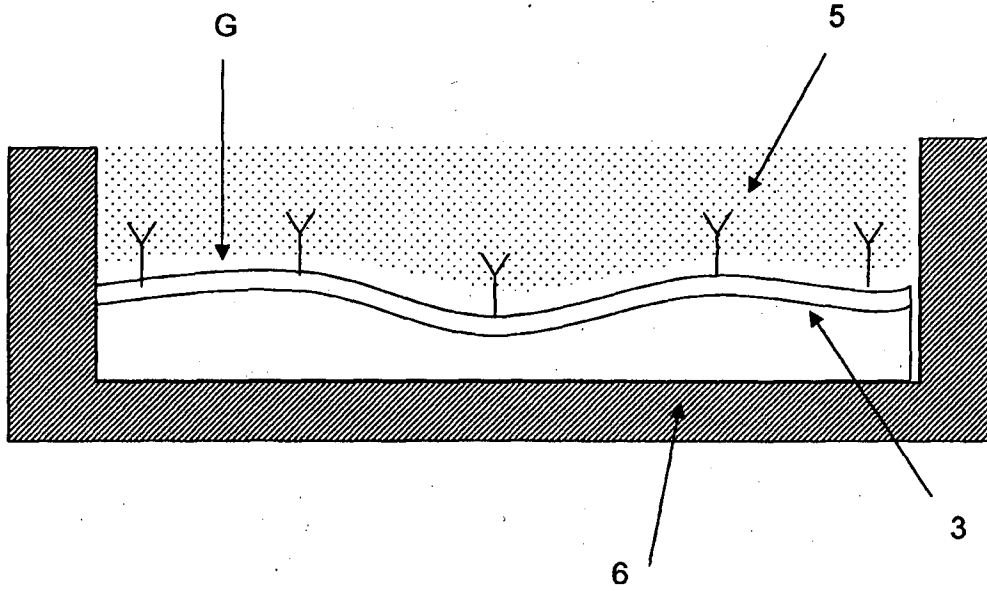


Figure 5

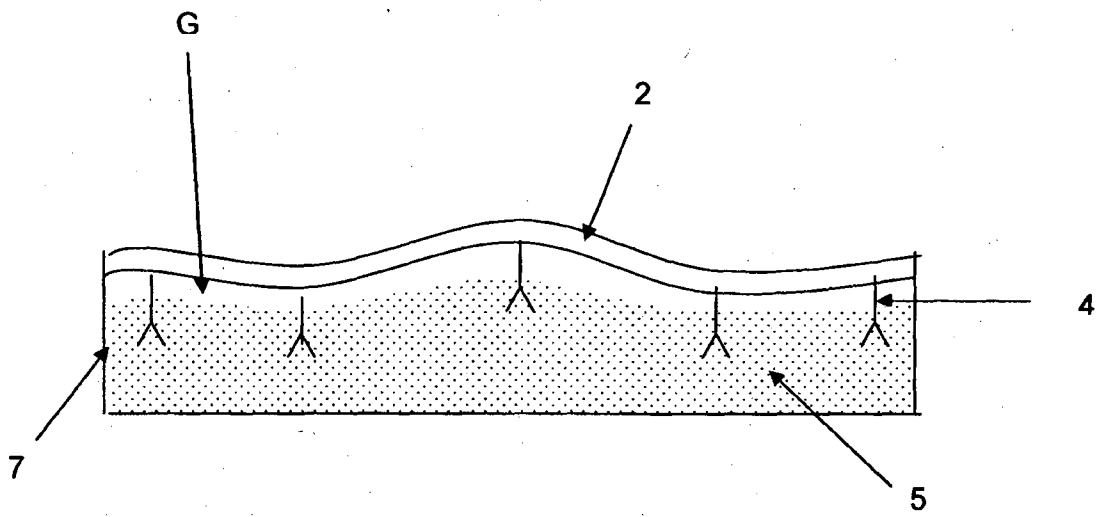


Figure 6

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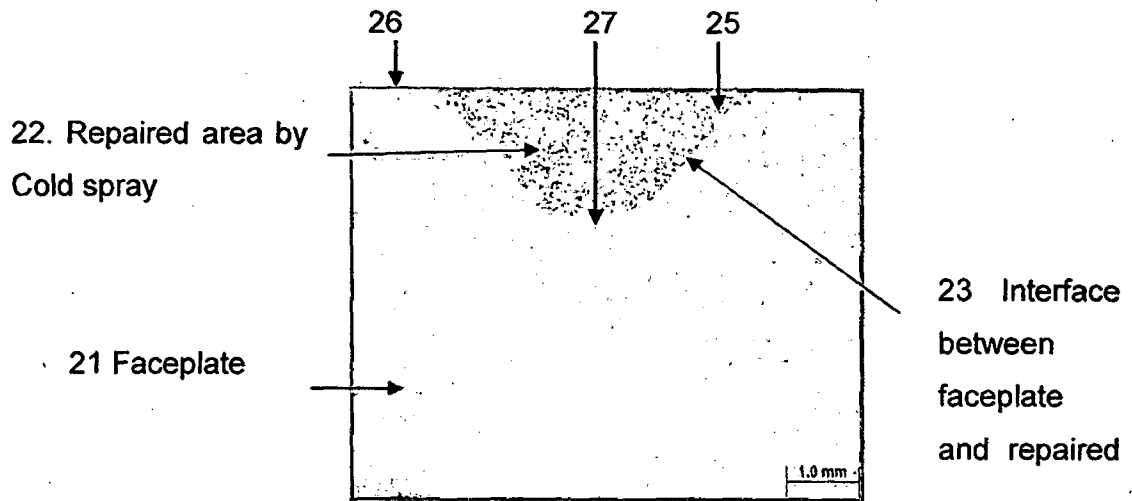


Figure 7

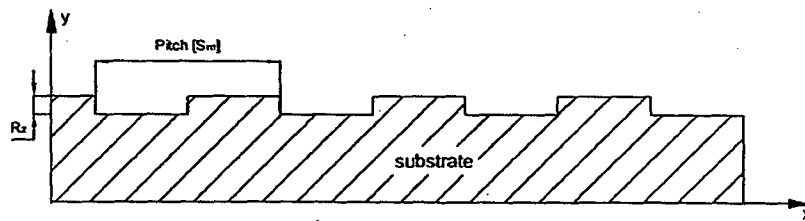


Figure 8

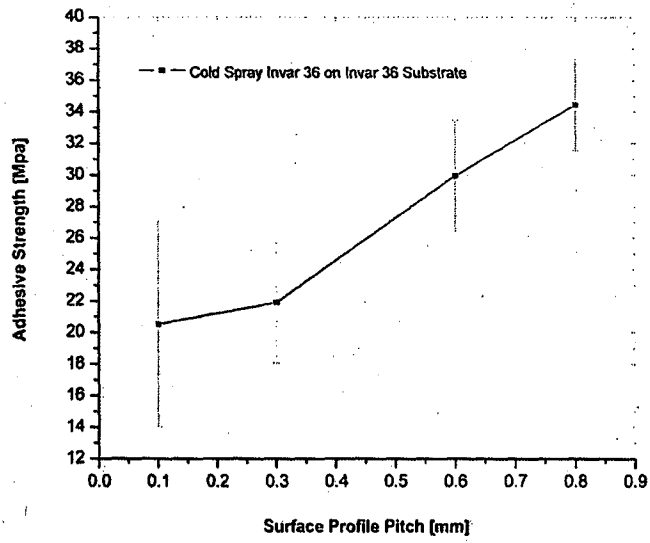


Figure 9

INTERNATIONAL SEARCH REPORT

International application No.

PCT/AU2013/000920

A. CLASSIFICATION OF SUBJECT MATTER

B29C 70/30 (2006.01) B21D 37/02 (2006.01) C23C 24/04 (2006.01) B21K 5/20 (2006.01) B05B 13/00 (2006.01)

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)

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)

EPODOC and WPI:IC/EC C23C24/04 and keywords (laid+ or lay+) & (thermal or coefficient or expansion), (cold w spray+) or (cold_gas w dynamic+) or (gas_dynamic 5d cold) or cold_spray+ or (kinetic+ w spray) or (low_temperature w spray+), composite face_plate, (aeronautical or aircraft or aerospace or boat+ or nautical or ship+)

Google with similar keyword(s).

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
	Documents are listed in the continuation of Box C	



Further documents are listed in the continuation of Box C



See patent family annex

* Special categories of cited documents:	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A" document defining the general state of the art which is not considered to be of particular relevance	"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"E" earlier application or patent but published on or after the international filing date	"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"&" document member of the same patent family
"O" document referring to an oral disclosure, use, exhibition or other means	
"P" document published prior to the international filing date but later than the priority date claimed	
Date of the actual completion of the international search 28 November 2013	Date of mailing of the international search report 28 November 2013
Name and mailing address of the ISA/AU AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA Email address: pct@ipaustralia.gov.au Facsimile No.: +61 2 6283 7999	Authorised officer Gregory Diven AUSTRALIAN PATENT OFFICE (ISO 9001 Quality Certified Service) Telephone No. 0262832992

INTERNATIONAL SEARCH REPORT

International application No.

C (Continuation).

DOCUMENTS CONSIDERED TO BE RELEVANT

PCT/AU2013/000920

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2007/0215677 A1 (FLOYD ET AL) 20 September 2007 abstract, paragraphs [0003], [0006], [0020]-[0028], [0030], claims	1, 2, 3, 6, 11, 12, 13, 16
X	US 5302414 A (ALKHIMOV ET AL) 12 April 1994 abstract; col 8 line 15- col 9 line 45; examples 1, 8; claims 1-6	1, 3, 11, 13
A	US 6759002 B1 (ENGWALL ET AL) 06 July 2004 abstract; col 5 line 34-col 8 line 13	1-21
A	US 7141191 B2 (ENGWALL ET AL) 28 November 2006 abstract; col 5 line 42- col 6 line 4, claims 1-16	1-21
A	US 6749002 B2 (GRINBERG ET AL) 15 June 2004 abstract; fig 6; col 2 line 23- col 3 line 12, col 6 lines 1-4	1-21
A	US 6759085 B2 (MUEHLBERGER) 06 July 2004 abstract; claims	1-21

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:
the subject matter listed in Rule 39 on which, under Article 17(2)(a)(i), an international search is not required to be carried out, including
2. Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:
3. Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a)

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

See Supplemental Box for Details

1. As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.
2. As all searchable claims could be searched without effort justifying additional fees, this Authority did not invite payment of additional fees.
3. As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:
4. No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:
1-21

Remark on Protest

- The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.
- The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.
- No protest accompanied the payment of additional search fees.

Supplemental Box**Continuation of: Box III**

This International Application does not comply with the requirements of unity of invention because it does not relate to one invention or to a group of inventions so linked as to form a single general inventive concept.

This Authority has found that there are different inventions based on the following features that separate the claims into distinct groups:

- Claims 1-21 are directed to a composite lay up tool, and process for producing the composite lay up tool, comprising a metal faceplate on a support member, wherein the faceplate has been formed by cold-gas dynamic spraying of particles of the metal onto a substrate and wherein the support member is compatible with the faceplate and comprises a casting of a ceramic composition. The feature of the support member is compatible with the faceplate and comprises a casting of a ceramic composition is specific to this group of claims.
- Claims 22-29 are directed to a castable ceramic composition comprising a binder and an aggregate component mixable with water to form a slurry ready for casting, wherein the aggregate component is one of, or a mixture of, fused silica and a particulate metal alloy, and the binder and aggregate component are present in the proportions 10-50 wt% and 90-50 wt% based on the dry weight of the composition. The feature of a castable ceramic composition comprising a binder and an aggregate component is specific to this group of claims.
- Claims 30-33 are directed to a process for producing a faceplate for a lay up tool, which process comprises cold-gas dynamic spraying of particles of a nickel-iron alloy onto a substrate to form a faceplate of the nickel-iron alloy on the substrate, the faceplate being of a thickness in the range 2 to 10 mm. The feature of a process comprising cold-gas dynamic spraying of particles of a nickel-iron alloy onto a substrate to form a faceplate of the nickel-iron alloy on the substrate is specific to this group of claims.
- Claims 34-36 are directed to a composite lay up tool comprising a support member and a faceplate that comprises an assembly of cold-gas sprayed particles of a nickel-iron alloy. The feature of a composite lay up tool comprising a support member and a faceplate that comprises an assembly of cold-gas sprayed particles of a nickel-iron alloy is specific to this group of claims.
- Claims 37-43 are directed to a process for depositing a metal on a faceplate of a lay up tool in which the faceplate is formed of a nickel-iron alloy, which process comprises preparing an area of a surface of the faceplate to effect cleaning and conditioning of the area to accept and adhere to cold-gas dynamic sprayed particles, and cold-gas dynamic spraying particles of the metal onto the cleaned and conditioned area, wherein the metal is a nickel-iron alloy selected to be compatible with the faceplate. The feature of a process comprising preparing an area of a surface of the faceplate to effect cleaning and conditioning of the area to accept and adhere to cold-gas dynamic sprayed particles is specific to this group of claims.

PCT Rule 13.2, first sentence, states that unity of invention is only fulfilled when there is a technical relationship among the claimed inventions involving one or more of the same or corresponding special technical features. PCT Rule 13.2, second sentence, defines a special technical feature as a feature which makes a contribution over the prior art.

When there is no special technical feature common to all the claimed inventions there is no unity of invention.

In the above groups of claims, the identified features may have the potential to make a contribution over the prior art but are not common to all the claimed inventions and therefore cannot provide the required technical relationship. The only feature common to all of the claimed inventions and which provides a technical relationship among them is a metal faceplate for a lay up tool where the faceplate has been formed by cold spraying.

However this feature does not make a contribution over the prior art because it is disclosed in:

US 6749002 B2 (Grinberg et al.) 15 June 2004

Form PCT/ISA/210 (Supplemental Box) (July 2009)

Supplemental Box

Therefore in the light of this document this common feature cannot be a special technical feature. Therefore there is no special technical feature common to all the claimed inventions and the requirements for unity of invention are consequently not satisfied *a posteriori*.

It is considered that there are 3 groups of inventions. The groups are:

Group 1 comprising claims 1-10 and 11-21 (1-21)

Group 2 comprising claims 22-29

Group 3 comprising claims 30-33, 34-36 and 37-43

Group 1 comprises claims relating to a composite lay up tool which has a substrate comprising a casting of a ceramic composition to which is attached a faceplate which has been formed by cold spraying.

Group 2 comprises claims to a castable composition.

Group 3 although similar to group 1 in being related to a faceplate for a lay up tool differs in that they specify that the faceplate is made of nickel-iron alloy and there is no disclosure of the substrate or that the substrate should comprise a castable ceramic composition.

Group 2 does not share any common features with group 3, nor does the castable ceramic composition of group 1 link with that of group 2 as there is no limitation of the ceramic in claims 1 or 11 of group 1.

Thus group 2 is considered to share no features in common with groups 1 and 3.

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/AU2013/000920

This Annex lists known patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document/s Cited in Search Report		Patent Family Member/s	
Publication Number	Publication Date	Publication Number	Publication Date
US 2007/0215677 A1	20 Sep 2007	None	
US 5302414 A	12 Apr 1994	EP 0484533 B1	25 Jan 1995
		US 5302414 A	12 Apr 1994
		US 5302414 B1	25 Feb 1997
		WO 9119016 A1	12 Dec 1991
US 6759002 B1	06 Jul 2004	CA 2201981 A1	08 Oct 1997
		CA 2283938 A1	12 Nov 1998
		CN 1168317 A	24 Dec 1997
		CN 1255076 A	31 May 2000
		EP 0807504 A1	19 Nov 1997
		EP 0807504 B1	02 Oct 2002
		EP 0981411 A1	01 Mar 2000
		US 5746553 A	05 May 1998
		US 6012883 A	11 Jan 2000
		US 6168358 B1	02 Jan 2001
		US 6759002 B1	06 Jul 2004
		WO 9850180 A1	12 Nov 1998
US 7141191 B2	28 Nov 2006	US 7141191 B2	28 Nov 2006
US 6749002 B2	15 Jun 2004	EP 1413642 B1	15 Mar 2006
		JP 2004143598 A	20 May 2004
		US 2004076807 A1	22 Apr 2004
		US 6749002 B2	15 Jun 2004
US 6759085 B2	06 Jul 2004	EP 1558400 A1	03 Aug 2005
		US 2003232132 A1	18 Dec 2003
		US 6759085 B2	06 Jul 2004
		WO 03106051 A1	24 Dec 2003

End of Annex

Due to data integration issues this family listing may not include 10 digit Australian applications filed since May 2001.

Form PCT/ISA/210 (Family Annex)(July 2009)