Method for fabrication of a fiber metal laminate.

Method for fabrication of a fiber metal laminate that comprises metal layers and in between said metal layers fiber reinforced resin layers, comprising a pressure treatment and a curing treatment of the fiber metal laminate, wherein the pressure treatment comprises the steps of:
- placing the first bag with the fiber metal laminate under a second bag or cover,
- drawing a vacuum in said first and second bag or cover, wherein
- the pressure treatment and the curing treatment of the fiber metal laminate are executed separate from each other.

Octrooi is verleend ongeacht het bijgevoegde resultaat van het onderzoek naar de stand van de techniek en schriftelijke opinie. Het octrooischrift komt overeen met de oorspronkelijk ingediende stukken.
Method for fabrication of a fiber metal laminate

The invention relates to a method for fabrication of a fiber metal laminate that comprises metal layers and in between said metal layers fiber reinforced resin layers, comprising placement of the fiber metal laminate in a first bag or cover for vacuuming the fiber metal laminate, followed by a curing treatment of the fiber metal laminate at elevated pressure.

Such a method is commonly used for the production of aircraft fuselage skin material, being assembled of thin aluminum sheets with in-between cross ply pre-impregnated glass fiber/epoxy resin layers. It is known to manufacture such material by laying up the individual layers and plies on or in a mould. Once the total stack has been assembled, the panel is vacuum bagged for curing. In this known method the curing process is performed in an autoclave, because of the temperature (120°C-175°C depending on the epoxy resin) and pressure (6-11 bars) required to obtain sufficient quality.

The necessity to use in the prior art an autoclave implies not only significant costs, but also a limitation in the maximum dimensions of the panels that can be manufactured according to this known method. The high pressure during the autoclave cure cycle according to this known manufacturing method is necessary to push the air bubbles (voids) out of the panel, or to make the voids collapse inside the panel during the curing cycle. This may necessitate that for certain fiber metal laminate panels the applied pressure may be as high as 11 bars.

It is an objective of the invention to provide a method for manufacturing a fiber metal laminate having less restrictions and disadvantages then the known method as discussed herein above.

It is a further objective of the invention to obviate the need of using an autoclave for the curing process and thus provide the possibility to increase the dimensions of the fiber metal laminate that is being manufactured.

The method for fabrication of a fiber metal laminate in accordance with the invention has to this end the features of one or more of the appended claims.
The invention is essentially characterized in that the pressure treatment comprises the steps of
placing the first bag with the fiber metal laminate under a second bag or cover, and
-drawing a vacuum in said second bag or cover,
and that the pressure treatment and the curing treatment of the fiber metal laminate are executed separate from each other.

It is remarked that WO2005/113213 teaches a double vacuum bag process for resin matrix composite manufacturing,
wherein a first vacuum bag (inner bag) is sealed to a tool plate with a vacuum port built into the tool plate inside the inner bag connected to a vacuum pump to draw a vacuum within the inner bag. A second bag designated as an outer bag is similarly situated external to the inner bag with a perforated tool disposed intermediate to the outer bag and the inner bag. Further, a second vacuum port provided intermediate to the outer bag and inner bag allows for the creation of a second vacuum atmosphere or environment.

In operating this known method according to WO2005/113213 a low-temperature ramp-and-hold period is applied to the processed laminate by introducing a vacuum into the outer bag at a higher vacuum than the vacuum introduced in the inner bag. This pulls the inner bag towards the outer bag, and does not compress the laminate. Since the laminate is not compacted by vacuum pressure, it remains loose and volatiles are free to escape by the vacuum suction from the inner bag vacuum pump during this stage. At the end of the low-temperature ramp-and-hold period the outer bag is purged to atmospheric pressure, while the vacuum within the inner bag is increased. This causes the inner bag to collapse to consolidate the laminate during the high temperature ramp-and-hold period of the two-step cycle that follows the low-temperature ramp-and-hold. In this known method the laminate, which does not comprise metal layers, can be placed in any heating chamber, such as an oven, a vacuum oven, a vacuum press, or an autoclave to apply the necessary heat for the cure cycle stages.

According to the invention it has been proven possible to apply a pressure treatment on a fiber metal laminate by placing the first bag with the fiber metal laminate under a second bag or cover, and by drawing a vacuum in said second bag or
cover, wherein the pressure treatment and the curing treatment of the fiber metal laminate are executed separate from each other. This avoids using a two-step temperature ramp-and-hold cycle for curing as is taught by WO2005/113213, according to which teaching this curing cycle requires to maintain an elevated pressure on the laminate by applying a vacuum in the second bag or cover during the low-temperature ramp-and-hold period. Contrary to the known prior art, according to the invention it is possible that the pressure treatment is executed separate and preferably prior to the curing treatment of the fiber metal laminate.

In the method of the invention it is further proven sufficient and preferable that during the curing treatment of the fiber metal laminate the vacuum of the fiber metal laminate in the first bag is maintained. This secures that during curing the elements of the laminate are maintained in position and that the return of pressed out air is prevented.

Summarized it can be said that in the method of the invention, all air that resides in the laminate is enabled to be permanently removed during the pressure treatment prior to the curing treatment. Once the removal of the air bubbles from the laminate is complete it suffices to maintain the vacuum of the fiber metal laminate in the first bag and to carry out then the curing treatment of the fiber metal laminate. During curing it is not necessary to apply any external pressure. It suffices that the laminate is cured while it is under vacuum. In this way, the laminate can be cured with only the atmospheric pressure applied to the vacuum bag in which the laminate is contained.

Since no additional pressure is necessary to be applied during curing on the laminate, the laminate can be cured in either an oven, or using heat blankets or heating devices above or inside a mould. This brings about the advantage that both the vacuum cycle and the cure cycle can be performed at the same location where the laminate has been assembled, without necessity to physically move the laminate to another location or to an autoclave. This makes it easier to manufacture also very large laminates.

The invention will now further be explained with reference to the drawing, showing in:
-figure 1 a setup of a fiber metal laminate placed on a mould;

-figure 2 the fiber metal laminate of figure 1 placed in a vacuum bag and under a second cover;

-figures 3-6 four different curing methods to be applied while the fiber metal laminate is in a vacuum bag;

-figure 7 a comparison of the tensile stress strain behavior of a conventionally manufactured (cross ply and unidirectional) laminate and a (cross ply and unidirectional) laminate manufactured according to the invention;

-figure 8 a comparison of the compressive stress strain behavior of a conventionally manufactured (cross ply and unidirectional) laminate and a (cross ply and unidirectional) laminate manufactured according to the invention;

-figure 9 a comparison of the interlaminar shear properties of a conventionally manufactured (cross ply and unidirectional) laminate and a (cross ply and unidirectional) laminate manufactured according to the invention;

-figure 10 a comparison of the fatigue properties of a conventionally manufactured (cross ply and unidirectional) laminate and a (cross ply and unidirectional) laminate manufactured according to the invention.

With reference to figure 1 a very schematic setup is shown of a laminate 1, which is assembled on top of a mould 2 prior to it being subjected to the processing steps of the method of the invention.

In figure 2 it is shown that the laminate 1 is placed in a first bag or cover 3 for vacuuming the fiber metal laminate 1, such that it can subsequently be subjected to a pressure treatment comprising the steps of placing the first bag 3 with the fiber metal laminate 1 under a second bag or cover 4, and drawing a vacuum in said second bag or cover 4 for executing a pressure treatment on the laminate 1. During this pressure treatment all air that resides in the laminate 1 is enabled to be removed prior to the curing treatment that can be executed thereafter. Once the removal of the air bubbles from the laminate 1 is complete it suffices to maintain the vacuum of the fiber metal laminate 1 in the first bag 3 and to carry out then the curing treatment of the fiber metal laminate 1.
Possible embodiments of curing treatments are respectively shown in the figures 3-6.

Figure 3 shows a curing treatment using an oven 5.

Figure 4 shows a curing treatment using the mould 2 as a heating device.

Figure 5 shows a curing treatment with a separate meeting device 6 above the mold 2.

Figure 6 shows a curing treatment with the application of a heating blanket 7.

Tensile properties

With reference to figure 7 it is shown that the laminate of the invention 7.3, 7.4 exhibits comparable tensile stress strain behavior in comparison with a conventionally manufactured laminate 7.1, 7.2. The laminates of this figure 7 are laminates having unidirectionally 7.1, 7.3 and cross ply 7.2, 7.4 oriented fibers. The conventionally manufactured laminate was subjected to a standard cure cycle at 120°C and 6 bars in an autoclave.

Compression properties

With reference to figure 8 it is shown that the laminate of the invention 8.3, 8.4 exhibits comparable compressive stress strain behavior in comparison with a conventionally manufactured laminate 8.1, 8.2. The laminates of this figure 8 are laminates having unidirectionally 8.1, 8.3 and cross ply 8.2, 8.4 oriented fibers. The conventionally manufactured laminate was subjected to a standard cure cycle at 120°C and 6 bars in an autoclave.

Interlaminar shear properties

With reference to figure 9 it is shown that the laminate of the invention 9.3, 9.4 exhibits comparable interlaminar shear behavior in comparison with a conventionally manufactured laminate 9.1, 9.2. The laminates of this figure 9 are laminates having unidirectionally 9.1, 9.3 and cross ply 9.2, 9.4 oriented fibers. The conventionally manufactured laminate was subjected to a standard cure cycle at 120°C and 6 bars in an autoclave.
Fatigue properties

With reference to figure 10 it is shown that the laminate of the invention 10.3, 10.4 exhibits comparable fatigue crack growth behavior in comparison with a conventionally manufactured laminate 10.1, 10.2. The laminates of this figure 10 are laminates having unidirectionally 10.1, 10.3 and cross ply 10.2, 10.4 oriented fibers. The conventionally manufactured laminate was subjected to a standard cure cycle at 120°C and 6 bars in an autoclave.
CONCLUSIES

1. Werkwijze voor het vervaardigen van een vezelmetaal laminaat (1) welke metaallagen en tussen genoemde metaallagen vezelversterkte harslagen omvat, omvattende het plaatsen van het vezelmetaal laminaat (1) in een eerste zak of afdekking (3) voor het vacuümeren van het vezelmetaal laminaat (1), gevolgd door een drukbehandeling en een uithardbehandeling van het vezelmetaal laminaat, met het kenmerk, dat de drukbehandeling de stappen omvat van het
   - plaatsen van de eerste zak (3) met het vezelmetaal laminaat (1) onder een tweede zak of afdekking (4), en
   - het trekken van een vacuüm in genoemde tweede zak of afdekking (4), en dat
   - de drukbehandeling en de uithardbehandeling van het vezelmetaal laminaat (1) gescheiden van elkaar worden uitgevoerd.

2. Werkwijze voor het vervaardigen van een vezelmetaal laminaat volgens conclusie 1, met het kenmerk, dat
   - de drukbehandeling uitgevoerd wordt gescheiden en voorafgaand aan de uithardbehandeling van het vezelmetaal laminaat (1).

3. Werkwijze voor het vervaardigen van een vezelmetaal laminaat volgens conclusie 1 of 2, met het kenmerk, dat gedurende de uithardbehandeling van het vezelmetaal laminaat (1) het vacuüm van het vezelmetaal laminaat in de eerste zak (3) gehandhaafd wordt.
Fig. 7

Fig. 8
SAMENWERKINGSVERDRAG (PCT)
RAPPORT BETREFFENDE NIEUWHEIDSONDERZOEK VAN INTERNATIONAAL TYPE

<table>
<thead>
<tr>
<th>IDENTIFICATIE VAN DE NATIONALE AANVRAGE</th>
<th>KENMERK VAN DE AANVRAGER OF VAN DE GEMACHTIGDE</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>NL 48335-VB</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Nederlands aanvraag nr.</th>
<th>Indieningsdatum</th>
</tr>
</thead>
<tbody>
<tr>
<td>2005667</td>
<td>11-11-2010</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Ingeroepen voorrangsdatum</th>
</tr>
</thead>
</table>

<table>
<thead>
<tr>
<th>Aanvrager (Naam)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Technische Universiteit Delft</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Datum van het verzoek voor een onderzoek van internationaal type</th>
<th>Door de Instantie voor Internationaal Onderzoek aan het verzoek voor een onderzoek van internationaal type toegekend nr.</th>
</tr>
</thead>
<tbody>
<tr>
<td>05-02-2011</td>
<td>SN 55628</td>
</tr>
</tbody>
</table>

I. CLASSIFICATIE VAN HET ONDERWERP (bij toepassing van verschillende classificaties, alle classificatiesymbolen opgeven)
Volgens de internationale classificatie (IPC)

| B32B37/10 | B32B15/08 | B29C70/44 |

II. ONDERZOCHTE GEBIEDEN VAN DE TECHNIEK

<table>
<thead>
<tr>
<th>Classificatiesysteem</th>
<th>Classificatiesymbolen</th>
</tr>
</thead>
<tbody>
<tr>
<td>IPC8</td>
<td>B32B</td>
</tr>
<tr>
<td></td>
<td>B29C</td>
</tr>
</tbody>
</table>

Onderzochte andere documentatie dan de minimum documentatie, voor zover dergelijke documenten in de onderzochte gebieden zijn opgenomen

III. GEEN ONDERZOEK MOGELIJK VOOR BEPAALDE CONCLUSIES (opmerkingen op aanvullingsblad)

IV. GEBREK AAN EENHEID VAN UITVINDING (opmerkingen op aanvullingsblad)

Form PCT/ISA 201 A (11/2000)
### ONDERZOEKSBAPORT BETREFFENDE HET RESULTAAT VAN HET ONDERZOEK NAAR DE STAND VAN DE TECHNIEK VAN HET INTERNATIONALE TYPE

#### A. CLASSIFICATIE VAN HET ONDERWERP

<table>
<thead>
<tr>
<th>INV.</th>
<th>B32B37/10</th>
<th>B32B15/08</th>
<th>B29C70/44</th>
</tr>
</thead>
</table>

Volgens de Internationale Classificatie van octrooien (IPC) of zowel volgens de nationale classificatie als volgens de IPC.

#### B. ONDERZOEkte GEbIEDEn VAN DE TECHNIEK

<table>
<thead>
<tr>
<th>Onderzochte minimum documentatie (classificatie gevolgd door classificatiesymbolen)</th>
</tr>
</thead>
<tbody>
<tr>
<td>B32B B29C</td>
</tr>
</tbody>
</table>

Onderzochte andere documentatie dan de minimum documentatie, voor dergelijke documenten, voor zover dergelijke documenten in de onderzochte gebieden zijn opgenomen

‘Tijdens het onderzoek geraadpleegde elektronische gegevensbestanden (naam van de gegevensbestanden en, waar uitvoerbaar, gebruikte trewoorden)’

EPO-Internal

#### C. VAN BELANG GEACHTe DOCUMENTEN

<table>
<thead>
<tr>
<th>Categorie</th>
<th>Geïsoleerde documenten, eventueel met aanduiding van speciaal van belang zijnde passages</th>
<th>Van belang voor conclusie nr.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>* alinea [0017] *</td>
<td>1-3</td>
</tr>
<tr>
<td></td>
<td>* alinea [0042] *</td>
<td></td>
</tr>
<tr>
<td></td>
<td>* figuur 4 *</td>
<td></td>
</tr>
<tr>
<td>A</td>
<td>EP 1 336 469 A1 (ALENIA AERONAUTICA SPA [IT]) 20 augustus 2003 (2003-08-20)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>* alinea [0010] *</td>
<td>1-3</td>
</tr>
<tr>
<td></td>
<td>* alinea [0020] *</td>
<td></td>
</tr>
<tr>
<td>X</td>
<td>EP 0 186 787 A2 (ASAH1 GLASS CO LTD [JP]) 9 juli 1986 (1986-07-09)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>* bladzijde 6, regel 4 - bladzijde 8,</td>
<td></td>
</tr>
<tr>
<td></td>
<td>regel 26 *</td>
<td>1-3</td>
</tr>
<tr>
<td></td>
<td>* figuur 5 *</td>
<td></td>
</tr>
</tbody>
</table>

☐ Verdere documenten worden vermeld in het vervolg van vak C.  
☒ Leden van dezelfde octrooifamilie zijn vermeld in een bijlage

* Speciale categorieën van aangehaalde documenten

**A** niet tot de categorie X of Y behorende literatuur die de stand van de techniek beschrijft

**O** in de octrooiaanvraag vermeld

**E** eerdere octrooiaanvraag, gepubliceerd op of na de indieningsdatum, waarin dezelfde uitvinding wordt beschreven

**L** om andere redenen vermelde literatuur

**C** niet-schriftelijke stand van de techniek

**P** tussen de voorrangsdatum en de indieningsdatum gepubliceerde literatuur

* ‘I’ na de indieningsdatum of de voorrangsdatum gepubliceerde literatuur die niet bezwavend is voor de octrooiaanvrage, maar wordt vermeld ter verheffing van de theorie of het principe dat ten grondslag ligt aan de uitvinding

‘X’ de conclusie wordt als niet nieuw of niet inventief beschouwd ten opzichte van deze literatuur

‘Y’ de conclusie wordt als niet inventief beschouwd ten opzichte van de combinatie van deze literatuur met andere geciteerde literatuur van dezelfde categorie, waarbij de combinatie voor de vakman voor de hand liggend wordt geacht

‘X’ lid van dezelfde octrooifamilie of overeenkomstige octrooipublicatie

Datum waarop het onderzoek naar de stand van de techniek van internationaal type werd voltooid

1 juni 2011

<table>
<thead>
<tr>
<th>Datum waarop het rapport van het onderzoek naar de stand van de techniek van internationaal type werd verkregen</th>
</tr>
</thead>
</table>

Verzendsdatum van het rapport van het onderzoek naar de stand van de techniek van internationaal type

Naam en adres van de instantie

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040
Fax. (+31-70) 340-3016

De bevoegde ambtenaar

Bataille, Laurent
<table>
<thead>
<tr>
<th>In het rapport genoemd octrooigeschrift</th>
<th>Datum van publicatie</th>
<th>Overeenkomend(e) geschrijven</th>
<th>Datum van publicatie</th>
</tr>
</thead>
<tbody>
<tr>
<td>EP 1336469 A1</td>
<td>20-08-2003</td>
<td>BR 0300588 A</td>
<td>08-09-2004</td>
</tr>
<tr>
<td></td>
<td></td>
<td>CA 2417072 A1</td>
<td>19-08-2003</td>
</tr>
<tr>
<td></td>
<td></td>
<td>JP 2003312590 A</td>
<td>06-11-2003</td>
</tr>
<tr>
<td></td>
<td></td>
<td>US 2003168555 A1</td>
<td>11-09-2003</td>
</tr>
<tr>
<td>EP 0186787 A2</td>
<td>09-07-1986</td>
<td>DE 3587118 D1</td>
<td>01-04-1993</td>
</tr>
<tr>
<td></td>
<td></td>
<td>DE 3587118 T2</td>
<td>09-06-1993</td>
</tr>
<tr>
<td></td>
<td></td>
<td>JP 1590728 C</td>
<td>30-11-1990</td>
</tr>
<tr>
<td></td>
<td></td>
<td>JP 61134249 A</td>
<td>21-06-1986</td>
</tr>
<tr>
<td></td>
<td></td>
<td>JP 63012786 B</td>
<td>22-03-1988</td>
</tr>
</tbody>
</table>
This opinion contains indications relating to the following items:

- ☑ Box No. I  Basis of the opinion
- ☐ Box No. II  Priority
- ☐ Box No. III  Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- ☐ Box No. IV  Lack of unity of invention
- ☑ Box No. V  Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- ☐ Box No. VI  Certain documents cited
- ☐ Box No. VII  Certain defects in the application
- ☐ Box No. VIII  Certain observations on the application

Examiner
WRITTEN OPINION

Box No. I  Basis of this opinion

1. This opinion has been established on the basis of the latest set of claims filed before the start of the search.

2. With regard to any nucleotide and/or amino acid sequence disclosed in the application and necessary to the claimed invention, this opinion has been established on the basis of:
   a. type of material:
      □ a sequence listing
      □ table(s) related to the sequence listing
   b. format of material:
      □ on paper
      □ in electronic form
   c. time of filing/furnishing:
      □ contained in the application as filed.
      □ filed together with the application in electronic form.
      □ furnished subsequently for the purposes of search.

3. □ In addition, in the case that more than one version or copy of a sequence listing and/or table relating thereto has been filed or furnished, the required statements that the information in the subsequent or additional copies is identical to that in the application as filed or does not go beyond the application as filed, as appropriate, were furnished.

4. Additional comments:

Box No. V  Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

   Novelty  Yes: Claims 1-3
             No:  Claims

   Inventive step  Yes: Claims
                    No: Claims 1-3

   Industrial applicability  Yes: Claims 1-3
                            No:  Claims

2. Citations and explanations

   see separate sheet

NL237B (July 2006)
Reference is made to the following documents:


D2 EP 1 336 469 A1 (ALENIA AERONAUTICA SPA [IT]) 20 augustus 2003 (2003-08-20)


Re Item V

Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

Independent claim 1

1 The present application does not meet the criteria of patentability, because the subject-matter of claim 1 does not seem to involve an inventive step.

1.1 Document D1 discloses a method for fabrication of a laminate (paragraph 0042 and figure 4) comprising placement of the layers in a bag (42) for vacuuming the laminate, followed by a pressure treatment and a curing treatment of the laminate, wherein the pressure treatment comprises the steps of:

- placing the bag with the laminate under a cover (66), and
- drawing a vacuum in said cover, wherein
- the pressure treatment and the curing treatment of the laminate are executed separate from each other.

The method of claim 1 only differs from that of D1 in that the laminate comprises metal layers and fiber reinforced resin layers in between.

The problem solved by the method according to D1 (cf. first sentence of paragraph 0017) is the same as that solved by claim 1, namely "to push the air bubbles out of the panel" (cf. description of the present application, page 1, lines 20-22), which is a usual step in the methods for manufacturing fiber metal laminates (cf. paragraph 0020 of document D2).
Moreover, the selection of the above mentioned materials for the laminate layers could only be regarded as inventive if it presented unexpected effects or properties in relation to the materials mentioned in D1. However, no such effects or properties are indicated in the application. Hence, no inventive step appears to be present in the subject-matter of claim 1.

1.2 For the same reasons as in above paragraph 1.1, the subject-matter of claim 1 does also not seem to be inventive over document D3 (see passages cited in the Search Report).

Dependent claims 2-3

2 Dependent claims 2-3 do not appear to contain any features which, in combination with the features of any claim to which they refer, meet the requirements of inventive step.