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(71) **Applicant:** PHILIPS ELECTRONICS N.V. [NL/NL]; Groenewoudseweg 1, NL-5621 BA Eindhoven (NL).

(71) **Applicant** *(for SE only)*: PHILIPS ELECTRONICS NORDEN AB [SE/SE]; Kottbygatan 5, Kista, S-164 85 Stockholm (SE).

(72) **Inventors:** GIJSBERS, Jan, Cornelis; Groenewoudseweg 1, NL-5621 BA Eindhoven (NL). VAN DE VEN, Adrianus, Johannes, Cornelis; Wielewaal 73, NL-5667 AB Geldrop (NL).

(74) **Agent:** BAKKER, Hendrik; Internationaal Octrooibureau B.V., P.O. Box 220, NL-5600 AE Eindhoven (NL).

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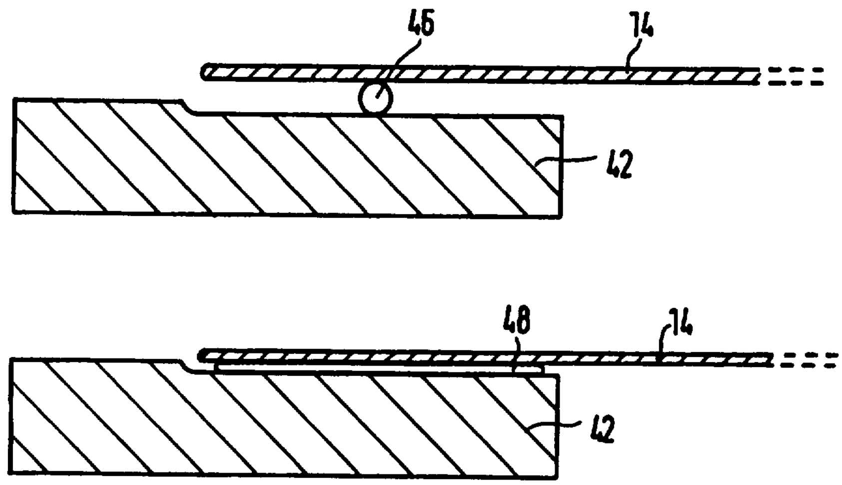
(54) **Title:** METHOD FOR THE VACUUMTIGHT SEALING OF A BERYLLIUM WINDOW TO A METAL SUBSTRATE

(57) **Abstract**

A method for the vacuumtight sealing of a thin (between 25 $\mu$m and 150 $\mu$m) beryllium X-ray window (14) to a metal substrate (42). The seal is realised by pressing an annular preform (46) of pure (99.99 %) aluminium of round cross section between the substrate (42) and the beryllium X-ray window (14) for a period of 20 s at a pressure of the order of magnitude of 50 N/mm$^2$, measured across the surface of the flattened aluminium, at a temperature of 500 °C.



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# Method for the vacuumtight sealing of a beryllium window to a metal substrate.

The invention relates to a method for the vacuumtight sealing of a thin beryllium window to a metal substrate.

A method of this kind is known from United States Patent Specification No. 4,431,709. According to the method described therein, several intermediate layers are provided between the window and the metal substrate. On the beryllium there is provided a first layer which can suitably adhere to the beryllium. This layer consists of, for example titanium; however, other, chemically related metals are also feasible. However, it is a common drawback of these metals that, generally speaking, their solderability is poor, so that they poorly adhere to the metal of the substrate, for example Kovar according to the known method. In order to solve this problem, above the first layer there is provided a layer which can be readily soldered, for example a nickel layer, but other readily solderable metals are also feasible. In order to prevent the metals of the said two layers from dissolving one into the other so that their adhesive effect is lost, the known method involves the use of a barrier layer, for example of vanadium, for keeping the first two layers separated. After deposition of these layers on the beryllium window, the window can be soldered to the substrate. The solder required for this purpose is a mixture of mainly copper and silver, it being necessary to heat the workpiece to 780 °C. However, if the beryllium window is heated to this temperature, the solder is liable to creep along the edges of said three layers so that it reaches the beryllium which then dissolves in the solder, thus degrading the reliability of the soldered seal.

Manufacturers and users of X-ray equipment, such as X-ray tubes or X-ray detectors, tend to utilize increasingly thinner beryllium windows because of their lower absorption for longwave X-rays. Known beryllium windows have a thickness of the order of magnitude of 150 $\mu$m (for example, see the cited United States Patent Specification which mentions a thickness of 250 $\mu$m). Nowadays a thickness of less than 100 $\mu$m, even as little as 25 $\mu$m is pursued. For these thicknesses said drawback of dissolving of the beryllium is even more pronounced. Moreover, a beryllium window of said small thickness tends to warp at the stated temperatures, so that appropriate vacuumtight sealing (for example, as required in X-ray tubes) cannot be achieved.

Moreover, in the known method for sealing beryllium windows the substrate must also have a thermal expansion coefficient which approximately equals that of beryllium, so that the choice of the metals and alloys for the substrate is limited.

It is an object of the invention to provide a method for sealing a thin beryllium window to a metal substrate in a reliable, vacuumtight manner.

To this end, the method of the invention is characterized in that an aluminium intermediate layer is formed in the plane of contact between the window and the substrate, the aluminium being provided in the form of a ring between the substrate and the window, after which the stack consisting of the substrate, the aluminium ring and the window is compressed by a pressure and at temperature sufficient to render the aluminium ductile.

The described steps produce a seal between the beryllium window and the substrate which is suitably vacuumtight also in the case of elevated temperatures of the order of 450 °C as required for the degassing of, for example an X-ray tube upon its evacuation. Furthermore, for this method of sealing the substrate and the window need not have the same thermal expansion coefficient. This is due to the fact that the method in accordance with the invention does not involve the temperatures occurring in the present state of the art; moreover, because of the ductile behaviour of the aluminium, a seal is realised which can readily take up displacements caused by different thermal expansions, so that no mechanical stresses endangering the quality of the seal will arise. The invention is based on the recognition of the fact that upon compression the oxide layer always present on the aluminium ring breaks open, so that the pure, non-oxidized aluminium comes into contact with the beryllium and with the substrate, so that strong and durable adhesion is achieved. Moreover, it may occur that not only the oxide layer present on the aluminium breaks open, but also that the oxide layers present on the beryllium and the substrate are broken open and pushed aside by the aluminium being displaced during compression.

In order to enhance the effect of the rupture of the oxide layer on the aluminium, a preferred embodiment of the invention is characterized in that the aluminium ring has a circular or elliptical cross-section. As is known, such a cross-section has a minimal circumference which is comparatively strongly enlarged by the compression.

Tests have demonstrated that suitably reliable adhesion is obtained when in conformity with a further step of the invention the purity of the aluminium is better than

99.9% by weight.

In conformity with a further step of the invention, the stack formed by the substrate, the aluminium ring and the beryllium window is compressed in an atmosphere which is poor in oxygen. This reduces the risk that oxygen from the environment forms a new oxide layer again immediately upon rupture of the oxide layers. It has been found that a mixture of nitrogen and hydrogen, notably with a ratio of 93% by volume of nitrogen and 7% by volume of hydrogen, offers suitable results in this respect.

A sufficiently high degree of ductility of the aluminium benefits the sealing process. To this end, in conformity with a further step of the invention the temperature at which the stack formed by the substrate, the aluminium ring and the window is compressed is between 450 °C and 550 °C.

Furthermore, the method of the invention is characterized in that the pressure at which the stack formed by the substrate, the aluminium ring and the window is compressed is between 30 $N/mm^2$ and 70 $N/mm^2$, measured across the surface of the flattened aluminium. Tests have demonstrated that optimum reliability of the seal is obtained when a pressure is selected from said range.

Further embodiments of the invention are described in the remaining dependent Claims.

These and other aspects of the invention will be apparent from and elucidated with reference to the embodiments described hereinafter.

In the drawings:

Fig. 1a is a sectional view of an X-ray tube comprising an X-ray window in accordance with the invention;

Fig. 1b is a sectional view of an X-ray detector comprising an X-ray window in accordance with the invention;

Fig. 2a shows an X-ray window in accordance with the invention prior to compression with the substrate;

Fig. 2b shows an X-ray window in accordance with the invention after compression with the substrate;

Fig. 3a is a detailed representation of an X-ray window in accordance with the invention prior to compression with the substrate;

Fig. 3b is a detailed representation of an X-ray window in accordance with the invention after compression with the substrate;

Fig. 4 is a graphic representation of the variation of the temperature and the pressure as a function of time during compression of the X-ray window with the substrate in accordance with the invention.

Fig. 1a shows an X-ray tube in which the X-ray window in accordance with the invention can be used. The tube comprises a housing 2 which encloses a vacuum space 4 and in which the other tube components, such as a filament 6 enclosed by a cathode structure 8, are accommodated. Electrons emitted by the filament are incident on the anode 10 with a high energy which is converted mainly into heat. A substantial part of this heat is dissipated by cooling water transported via the ducts 12. In the anode 10 X-rays are generated and emanate from the tube via the X-ray window 14. This window should be as thin as possible so as to minimize the X-ray absorption; however, its thickness must be sufficient to withstand permanently the atmospheric pressure of the ambient air. This must so in various operating conditions, for example fluctuating temperatures of the tube and the window. Severe requirements are thus imposed as regards the seal between the window and the substrate, being the tube housing in this case.

Fig. 1b shows an X-ray detector in which the X-ray window in accordance with the invention can be used. The detector comprises a housing 20 which is provided with an entrance window 14. The housing encloses a space 22 which contains a discharge gas and which accommodates further detector components, such as an anode wire 24 which is insulated from the metal housing 20 by means of insulators 26. Incident X-rays 28 cause ionization of the discharge gas 22, so that a charge pulse is received by the anode wire 24, said pulse being processed by processing equipment (not shown) connected to output 30. The input window 14 should be as thin as possible so as to minimize the X-ray absorption; however, its thickness must be sufficient to ensure suitable gastight sealing in various operating conditions, for example fluctuating temperatures. Severe requirements are thus imposed as regards the seal between the window and the substrate, being the detector housing in this case.

Figs. 2a and 2b show a part 40 of a housing of an X-ray tube prior to (Fig. 2a) and after (Fig. 2b) compression of the window with the substrate which is in this case formed by the edge 42 of the housing 40. The part 40 of the housing has a conical shape in which there is recessed an opening 44 which is bounded by the edge 42. The vacuumtight seal must be realised between this edge and the beryllium window 14. To this end, aluminium in the form of a ring 46 of round cross-section is arranged between the substrate 42 and the window 14. After compression, the ring 46 has been flattened so as to form a ring 48 of elongate cross-section as will be described in detail hereinafter with reference to the Figs. 3a and 3b.

Figs. 3a and 3b show parts of the Figs. 2a and 2b, respectively, which are denoted therein by circular areas 50. Notably Fig. 3b clearly shows the elongate shape of the flattened ring 48. In order to achieve suitable sealing between the window 14 and the substrate 42, a round beryllium window 14 having a diameter of 23 mm and a thickness of 75 $\mu$m is assumed. The substrate 42 (i.e. the housing of the X-ray tube) may consist of copper or the copper-nickel alloy "Monel-400" or the nickel-iron alloy "NiFe-75" (DIN designation:RNi-5, materialnumber 2.4596); notably the latter two materials have a thermal expansion coefficient which approximates that of beryllium, so that temperature variations will not lead to undesirable material stresses. The round opening 44 has a diameter of 18 mm, whereas the outer circle of the edge 42 has a diameter slightly larger than 23 mm. Between the window 14 and the substrate 42 there is arranged an aluminium ring 46 having a diameter of 20 mm and a preferably round cross-section of a diameter of 0.7 mm and a material purity of 99.99% (by weight). Prior to the further treatment, the aluminium ring is degreased by immersion in hexane and subsequently in ethanol, after which further impurities are removed by heating the ring to 350 °C for 10 minutes. The stack formed by the window 14, the ring 46 and the substrate 40, 42 is heated to a temperature of 500 °C, for example in a furnace in which a pressure can still be exerted on the stack. In the furnace an atmosphere which is low in oxygen is maintained, for example a mixture of 93% $N_2$ and 7% $H_2$ (by volume). This atmosphere has the advantage that any residual $O_2$ (for example, caused by leakage) is bound by $H_2$. At said temperature of 500 °C the aluminium is sufficiently ductile so as to be deformed by a comparatively small force. A compressive force of 9 kN is exerted, corresponding to a pressure of 56 N/mm$^2$ for the above dimensions. This force is applied so that it gradually increases during 0.2 s, after which it is maintained for approximately 20 s. Subsequently, the pressure is reduced to zero in the course of some tens of seconds during which the temperature is allowed to decrease by gradual cooling. The described variation of pressure (p) and temperature (T) is graphically shown in Fig. 4 in which the temperature variation is denoted by a solid line and the pressure variation by a dashed line, both as a function of time (t).

It has been found that the above treatment provides sufficiently strong and reliable sealing of the beryllium window to the substrate.

CLAIMS:

1. A method for the vacuumtight sealing of a thin beryllium window (14) to a metal substrate (42),
characterized in that
* an aluminium intermediate layer (48) is formed in the plane of contact between the window (14) and the substrate (42), the aluminium being provided in the form of a ring (46) between the substrate (42) and the window (14),
* after which the stack consisting of the substrate (42), the aluminium ring (46) and the window (14) is compressed by a pressure and at a temperature sufficient to render the aluminium ductile.

2. A method as claimed in Claim 1, characterized in that the aluminium ring (46) has a circular or elliptical cross-section.

3. A method as claimed in Claim 1, characterized in that the purity of the aluminium is better than 99.9 % by weight.

4. A method as claimed in Claim 1, characterized in that the stack formed by the substrate (42), the aluminium ring (46) and the window (14) is compressed in an atmosphere which is poor in oxygen.

5. A method as claimed in Claim 4, characterized in that the atmosphere which is poor in oxygen consists of a mixture of nitrogen and hydrogen.

6. A method as claimed in Claim 1, characterized in that the temperature at which the stack formed by the substrate (42), the aluminium ring (46) and the window (14) is compressed between 450 °C and 500 °C.

7. A method as claimed in Claim 1, characterized in that the pressure at which the stack formed by the substrate (42), the aluminium ring (46) and the window (14) is compressed is between 30 $N/mm^2$ and 70 $N/mm^2$, measured across the surface of the flattened aluminium (48).

8. A method as claimed in Claim 7, characterized in that the pressure whereby the stack formed by the substrate (42), the aluminium ring (46) and the window (14) is compressed gradually increases in a period of time of between 0.1 s and 5 s.

9. A method as claimed in Claim 1, characterized in that the beryllium window (14) has a thickness of between 25 $\mu$m and 150 $\mu$m.

10. A method as claimed in Claim 1, characterized in that the substrate (42) consists of copper or a copper-nickel alloy or a nickel-iron alloy.

11. An assembly consisting of a beryllium window (14) and a metal mount, exclusively aluminium being present in the plane of contact between the window and the material of the mount.

12. An assembly as claimed in Claim 11, characterized in that the purity of the aluminium is better than 99.9% by weight.

13. An X-ray component, such as an X-ray tube or an X-ray detector, which is provided with a beryllium window (14) on a metal substrate (42) and in which exclusively aluminium is present in the plane of contact between the window and the substrate.

14. An X-ray component, such as an X-ray tube or an X-ray detector as claimed in Claim 13, provided with a beryllium window (14) in which the aluminium has a purity better than 99.9% by weight.

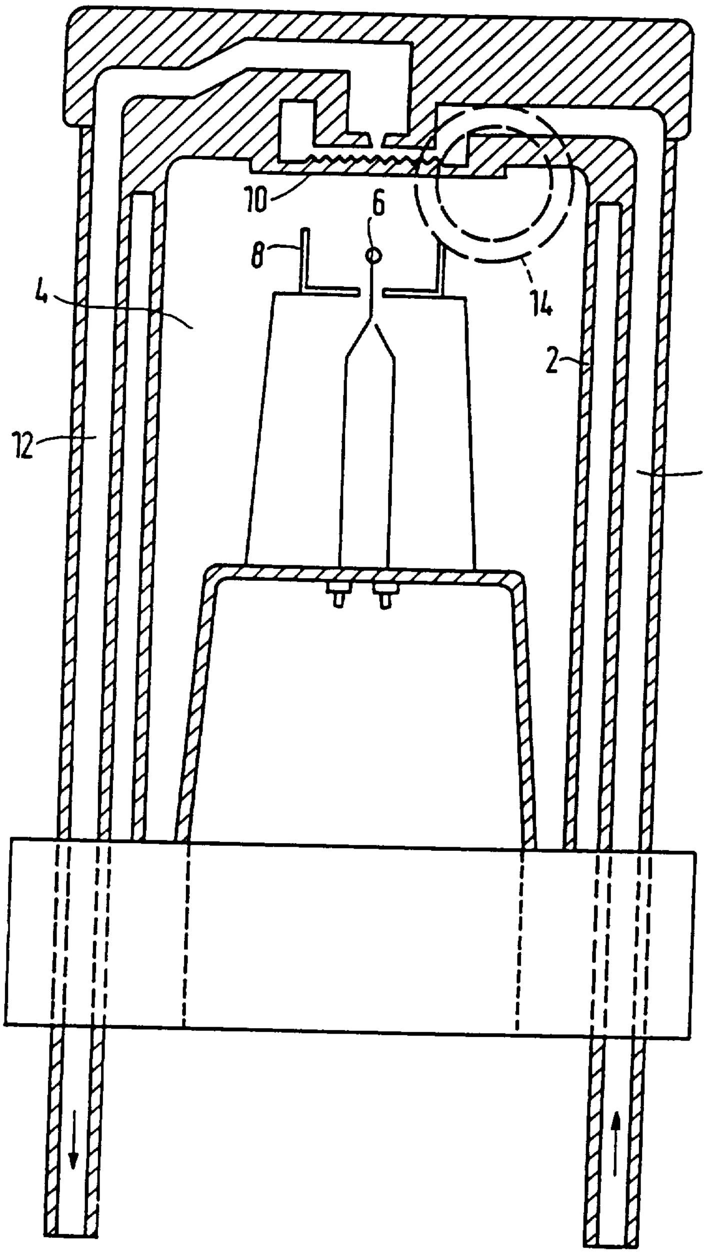


FIG.1a

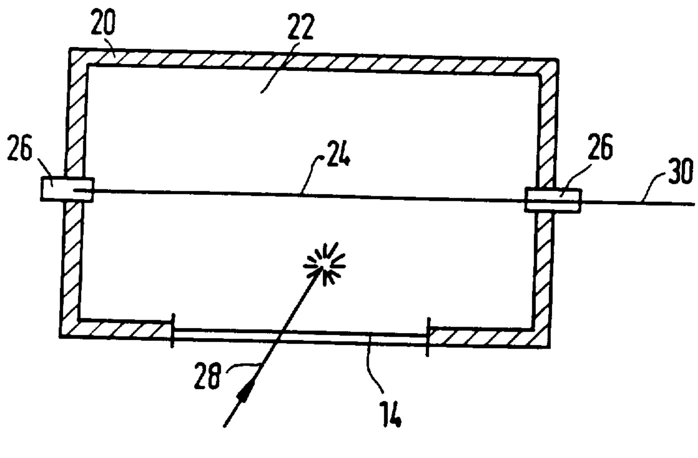


FIG.1b

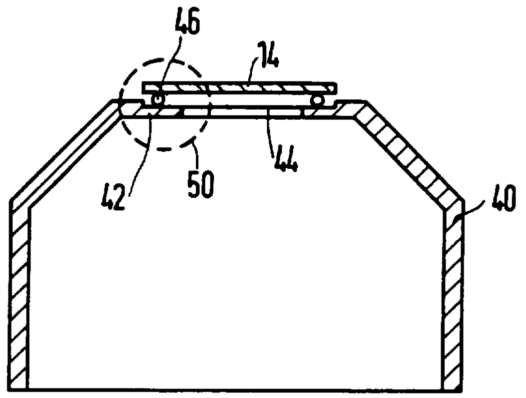


FIG.2a

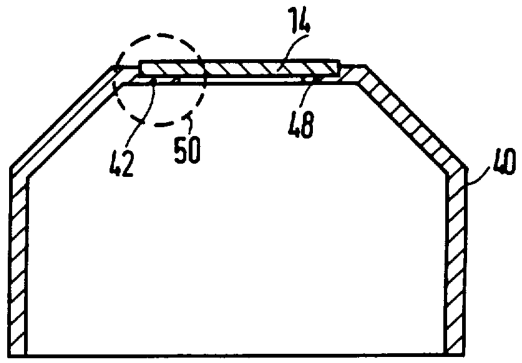


FIG.2b

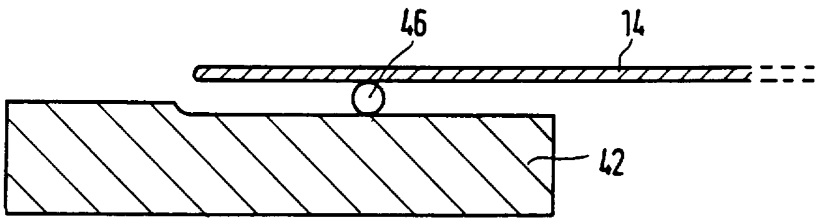


FIG.3a

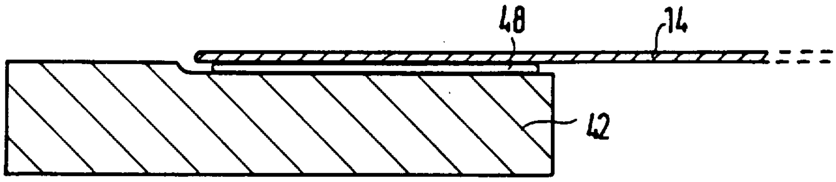


FIG.3b

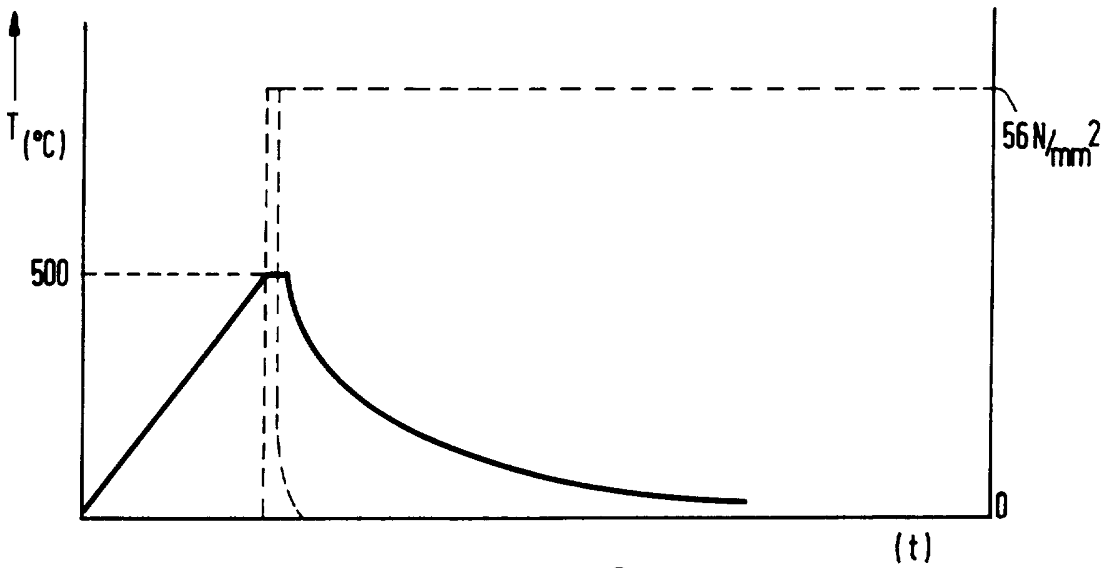


FIG.4

# INTERNATIONAL SEARCH REPORT

International application No.
PCT/IB 95/00948

**A. CLASSIFICATION OF SUBJECT MATTER**

IPC6: B23K 1/19, B23K 35/28 // H01J 35/18

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

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Minimum documentation searched (classification system followed by classification symbols)

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**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|---|---|---|
| X | Patent Abstracts of Japan, Vol 15,No 6, M-1066, abstract of JP, A, 2-258165 (RIGAKU CORP), 18 October 1990 (18.10.90)<br>-- | 1-14 |
| A | Patent Abstracts of Japan, Vol 12,No 328, E-654, abstract of JP, A, 63-91940 (MATSUSHITA ELECTRIC IND CO LTD), 22 April 1988 (22.04.88)<br>-- | 1-14 |
| A | EP 0699498 A1 (ALUMINUM DESIGN CORPORATION), 6 March 1996 (06.03.96), figure 1<br>--<br>-------- | 1-14 |

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

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01/04/96

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|---|---|---|---|
| EP-A1- 0699498 | 06/03/96 | NONE | |

Form PCT/ISA/210 (patent family annex) (July 1992)