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D E C I S I O N
of 18 December 1995

Case Number: T 0869/93 - 3.4.1

Application Number: 87302786.6

Publication Number: 0241204

IPC: H01L 21/20

Language of the proceedings: EN

Title of invention:
Method for forming crystalline deposited film

Applicant:
CANON KABUSHIKI KAISHA

Opponent:
-

Headword:
Single crystal growth/CANON II

Relevant legal provisions:
EPC Art. 56

Keyword:
"No enabling disclosure in prior document - novelty (yes)"
"Inventive step (yes; after amendment)"

Decisions cited:
T 0206/83, T 0081/87

Catchword:
-

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Case Number: T 0869/93 - 3.4.1

D E C I S I O N
of the Technical Board of Appeal 3.4.1
of 18 December 1995

Appellant: CANON KABUSHIKI KAISHA
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Ohta-ku
Tokyo (JP)

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Decision under appeal: **Decision of the Examining Division of the European Patent Office dated 6 May 1993 refusing European patent application No. 87 302 786.6 pursuant to Article 97(1) EPC.**

Composition of the Board:

Chairman: G. D. Paterson
Members: H. J. Reich
R. K. Shukla

Summary of Facts and Submissions

I. European patent application No. 87 302 786.6 (publication No. 0 241 204) was refused by a decision of the Examining Division.

II. The reason given for the refusal was that the subject-matter of Claims 1 of the main, first and second auxiliary requests filed on 2 March 1993 do not meet the requirements of Articles 52(1) and 56 EPC having regard to documents:

D1: Patent Abstracts of Japan, vol. 10, No. 99, (E-396), 16 April 1986 and JP-A-60/241222;
D3: British Journal of Applied Physics, vol. 18, No. 10, 1967, pages 1357 to 1382;
D4: US-A-3 620 833; and
D5: Philips Technical Review, vol. 41, No. 2, 1983/84, pages 60 to 69.

The reasons for the refusal of the application can be summarised as follows:

Document D4 is the most relevant prior art for Claims 1 of all requests and teaches (Figures 3 to 4 and column 2, lines 10 to 60 - in particular lines 33 to 39 -) that the relatively small nucleation sites (formed by imprinting a nucleating agent on a SiO_2 surface or by providing indentations or a selective damage of the SiO_2 layer) supports, under selected conditions of deposition, the formation and growth of one single nucleus only, since otherwise two or more crystals would have formed. It is also clear to the skilled person that under the conditions disclosed in document D4, the nucleation sites 10 have a larger nucleation density than the remainder of the SiO_2 layer 4, as crystals are

only formed at the nucleation sites. Document D3, page 1379, section 8 makes a skilled person aware of the fact that it is necessary to limit nucleation to a single nucleus and allow only this to grow. Thereby the skilled person's attention is drawn to document D4 which discloses a number of methods of limiting nucleation to a single (stable) nucleus at each nucleation site. Thus, the subject-matter of Claim 1 of the main request essentially differs from the disclosure in document D4 in that said crystals are nucleated and grown with a special vapour deposition process not explicitly disclosed in document D4; i.e. by vapour deposition using a gaseous starting material and a gaseous halogenic oxidising agent which react to form the species to be deposited. Having regard to such redox reaction the objective problem can be described as providing a deposition method which is suitable for nucleating and growing crystals at the selected nucleation sites at reduced temperatures ($T<1000°C$); see the description pages 19, 47 and 48. Since advantages of lowering processing temperatures are well known in the art, the recognition of the objective problem does not involve inventive skills. The deposition process disclosed in document D1 appears to be entirely suitable for solving the above objective problem, since it is clear to a skilled person that the halogen compound gases used in this method will oxidise straight chain silane at low temperatures to form a Si precursor which will be deposited. Thus, it would be obvious for the skilled person to substitute the deposition process of document D1 in the method of forming single crystals disclosed in document D4.

The subject-matter of Claim 1 of the **first auxiliary** request specifies silicon nitride material as deposition surface areas, silicon oxide as adjacent deposition surface area and silicon as deposited material. The use

of SiO_2 as the non-nucleation surface and of Si as the deposited material are disclosed in document D4, so that it would be obvious to use - in addition to the deposition process disclosed in document D1 - Si_3N_4 as alternative material for the nucleation sites in the method according to document D4 in view of the teaching in Figure 10 of document D5 that the nucleation density of Si on Si_3N_4 is larger than on SiO_2. Claim 1 of the **second auxiliary** request comprises additionally the feature that the deposition surface areas are established by lithographic patterning. Since lithographic patterning is well known in the art, also the subject-matter of Claim 1 of the second auxiliary request lacks an inventive step.

III. The Appellant lodged an appeal against this decision. In the Statement of Grounds of Appeal the Appellant filed a new main request which specified inter alia that the deposition surface areas are "of a different composition from that of the adjacent deposition surface area". Furthermore oral proceedings were auxiliarily requested.

IV. In a communication pursuant to Article 110(2) EPC dated 1 December 1994 the Board informed the Appellant of its provisional view that in Claim 1 of the main request the expression "gaseous **halogenic** oxidizing agent" required to be amended to read gaseous **halogen** oxidising agent" in order to avoid a novelty objection under Article 54(3) EPC based on document

D7: EP-A-0 244 081.

Furthermore, the Appellant was informed that in the embodiment disclosed in document D4, column 2, lines 10 to 36 nucleation sites having a relatively high nucleation density have a diameter of about 10^{-1} µm, which is therefore in the range of the examples

disclosed in the invention under appeal. In the Board's view, therefore, this embodiment hinted at the claimed size of the deposition surface areas. The Appellant was also informed that the use of the deposition process disclosed in document D1 in the method according to document D4 appeared not to produce any surprising effects. On the basis of the Appellant's submissions explaining the gist of the present invention, the Board proposed amendments to the main request in order to overcome objections under Articles 56, 84 and 123(2) and Rules 27(1)(b) and (c) EPC. In particular, a more specific definition was suggested in Claim 1 of the materials of the deposition surface area and of the deposited material which may epitaxially grow on the former.

V. In reply to the Board's communication the Appellant filed an amended main request on 10 February 1995.

Following consultations by telephone between the Rapporteur and the Appellant's representative on 21 June 1995 and 12 September 1995 wherein further amendments were discussed, the Appellant now requests that a patent be granted on the basis of the following main request:

Claims: 1 to 19 filed on 10 February 1995

Description: Pages 1 to 5 and 7 to 14, filed 9 February 1995 with the amendments on pages 1 and 7 requested on 21 June 1995; page 6, filed 10 February 1995;
(original page 15 cancelled)
original pages 16 to 77 with the amendments on page 42 requested on 9 February 1995 and on page 76 requested

on 21 June 1995, and with the amendment of the term "halogenic" into "halogen" throughout the description as requested on 12 September 1995

Drawings: Original sheet 1/29 to 29/29.

VI. Claim 1 of the main request reads as follows:

"1. A method for forming a crystalline material by deposition, comprising:

establishing at a free surface of a substrate a plurality of deposition surface areas (2; 6; 12; 6A; 9A; 12A) each being of a different composition from that of the adjacent deposition surface area (5; 11; 18; 20; 11A) and having under chosen conditions of deposition a property of nucleating a crystal forming material at a higher nucleation density than that of the adjacent deposition surface area and each of the deposition surface areas being at spaced apart locations on the substrate separated by said adjacent deposition surface areas; and

depositing said crystal forming material on the exposed free surface of the substrate so that said crystal forming material nucleates selectively on said deposition surface areas;

characterised in that:

(a) said deposition surface areas have surface properties which exclude an epitaxial growth of a single crystal of said crystal forming material;

(b) each of said deposition surface areas is limited to a size such that said crystal forming material forms on each deposition surface area under said chosen conditions of deposition one single nucleus of supracritical size (Figure 1) which grows into one single crystal; and

(c) the deposition of said crystal forming material is performed by introducing a gaseous starting material and a gaseous halogen oxidizing agent, having a property of oxidative action on said starting material, into a reaction chamber containing said substrate, whereby upon mutual contact said starting material and said agent interact to form a plurality of active species, at least one of these active species providing a source of said crystal forming material."

Claims 2 to 19 are dependent on Claim 1.

VII. In support of his requests the Appellant made essentially the following submissions:

(a) In the first embodiment disclosed in document D4, Figures 1 to 7 and column 1, line 62 to column 2, line 20 the deposition surface areas of high nucleation density are not formed by patterning a film on a substrate surface but by a printed-on nucleating agent. However this method is not disclosed in terms that are sufficiently clear and complete for it to be carried out, and the method would not work as disclosed. Having regard to the state of the fabrication technology at the filing date of document D4, the disclosure of document D4, should be regarded at best as based on isolated and non-reproducible results. Mechanical printing tools printing nucleation sites in the disclosed tenth of micron range (D4, column 2, lines 35 and 36) were

unavailable in 1966 and even today. The size of the nucleation site formed on the low nucleation density surface is critical to the obtaining of reliable results. Pushing tips into a silicon dioxide surface layer to provide indentations would not work reliably and is not compatible with integrated circuit technology. In the creation of deposition sites by electron bombardment, deposition of a single nucleus is not to be expected. Moreover, the nucleation agents disclosed in document D4 would decompose at the silicon deposition temperatures.

(b) In the second embodiment disclosed in document D4 at Figures 8 to 13 and column 3, lines 18 to 46, crystallites 16 are formed at random positions on the SiO_2-film rather than being formed at pre-selected nucleation sites. The actual randomly distributed locations have to be determined by scanning with a light beam and recording the resulting data in a computer (D4, column 3, lines 54 to 60). The purpose of the windows in the second embodiment of document D4 is to create sites where rapid epitaxial deposition of silicon reduces the formation of Si-crystallites on SiO_2 outside the windows. These windows provide locations for continued epitaxial growth, not selective nucleation and growth of new crystallites. Up to the present invention there was no reliable method of bringing about selective deposition of crystalline material at controlled nucleation sites on an amorphous substrate.

(c) The deposition process disclosed in document D1 is wholly concerned with the production of amorphous silicon. There is not a single reference in document D1 to the production of crystalline or

polycrystalline silicon. A skilled person would connect the low substrate temperature of 225°C in all examples of document D1 with the production of amorphous silicon. It would not be obvious that crystalline films could be produced at a low substrate temperature of 250°C as in the example 3 of the present application at page 75, line 9 and 14 to 23. The fact that a skilled person is capable of modifying the deposition of parameters of document D1 so that a crystalline film would grow epitaxially, is a presumption which is not supported by any document in the case and is based on impermissible hindsight.

(d) Although Claim 1 of the main request relies on feature (c) concerning the deposition process for novelty over document D7, all features - i.e. (a) concerning the size of the deposition surface areas, (b) concerning the composition of the deposition surface areas and also (c) - are relevant to inventive step; see the grounds of appeal pages 7 and 16.

(e) In referring to document D3, page 1379, section 8, the Examining Division has taken an isolated sentence out of context. The only experimental ways suggested in document D3 for obtaining a single crystal on an amorphous or polycrystalline substrate are based on an adjustment of the topography of the substrate by slip steps, etch patterns or indentations. Such methods are irrelevant to the present invention.

(f) Although document D5 discloses values for the nucleation densities of SiO_2 and Si_3N_4, the prior art does not hint at trying Si_3N_4 as nucleation site material (i.e. in the "deposition surface area") on

SiO_2 as the "adjacent deposition surface area". Furthermore, none of the methods disclosed in document D4 is suitable for imprinting small areas of a refractory material such as Si_3N_4. The benefits of lithographic patterning were generally known at the filing date of document D4. The different approaches of nucleation adopted in document D4 indicate that the skilled person was not aware of defining selective nucleation sites photolithographically as to size and position until the present invention.

Reasons for the Decision

1. Amendments and support in the description.

1.1 The subject-matter of Claim 1 of the main request comprises the characteristics of original Claims 1, 2, 4, 13, 15, 27 and of the original description page 30. The subject-matter of dependent Claims 2 to 19 is respectively disclosed in the original application documents as follows: Figures 20, 21 and 22; Figures 2, 6 and 8; Claim 15; Figure 2 and page 32 lines 25 and 26; Claim 14; Claim 20; Claim 16; Figures 10 to 13, 24 and 25; Figures 4C and 6C; Claim 19; Claim 22; Claim 23; Claim 24; Claim 25; Claim 26; Claim 28; Claim 29 and Claim 30.

1.2 In the Board's view, a skilled person is able to recognise that all examples for "crystal forming material" combined with the examples for the related "deposition surface" which examples are disclosed in the original description of the application in suit, have one property in common; namely, that the deposition surface has properties such that the corresponding

crystal forming material does not grow epitaxially into a single crystal onto the deposition surface. Therefore, amended Claim 1 including feature (a) satisfies Article 123(2) EPC. In view of the large variety of examples of materials for the deposition surface and the crystals, amended Claim 1 is supported by the description in the sense of Article 84 EPC. Feature (a) excludes from the subject-matter of Claim 1 combinations of crystal forming materials and related deposition surfaces, wherein the deposited material grows epitaxially on the deposition surface into a single crystal. However, the Board regards it necessary to note that in the following parts of the decision, feature (a) is regarded as a positive claim feature characterising a property which is inherent to the crystal forming material and to the related deposition surface claimed.

2. *Novelty - Claim 1 - Main request*

2.1 Document D4 discloses a "first embodiment" in Figures 1 to 7 and column 1, line 62 to column 3, line 17 and a "second embodiment" in Figures 8 to 13 and column 3, lines 18 to 60.

As to the "first embodiment", as summarised in paragraph VII-b above, the Appellant has argued that document D5 does not contain an "enabling disclosure" with respect to this embodiment. For enabling the growth of one single crystal on one nucleation site, document D4 discloses means for imprinting a nucleating agent or for mechanically damaging a substrate surface within an area of a diameter "in order of a tenth of a micron" (see D4, column 2, line 35). Means for operating in accordance with such small diameters are still not available today. Surface damage by an electron beam would not lead for a deposition of one single nucleus within one damaged area. Therefore, single crystal

growth cannot reliably be obtained by the disclosed measures of the first embodiment. The Board has at present no reason to doubt the Appellant's submissions in this respect. In accordance with the well-established case law of the Boards of Appeal (see in particular Decision T 206/83, OJ EPO 1987, 5, and T 81/87 OJ EPO 1990, 250 in order to destroy the novelty of a subsequently claimed invention, a prior disclosure must enable a skilled person to carry out that claimed invention. The Board is satisfied by the evidence filed by the Appellant in the present case that the disclosure of the "first embodiment" of document D5 would not enable a skilled person to carry out the invention claimed in Claim 1 of the main request.

2.2 As to the "second embodiment" of document D4, column 3, lines 18 to 60, in particular at lines 41 to 46, this describes a method of deposition including the steps defined in the precharacterising part of Claim 1, as agreed to by the Appellant; see also to the present introductory part of the description. In the Board's view, Claim 1 is novel over the "second embodiment" for the following reasons: Feature (a) is not disclosed in document D4 for the following reason: The windows formed in column 3, lines 18 to 46, in particular column 3, lines 41 to 46 expose as "deposition surface area" the surface of substrate 1 which consists - according to document D4, column 2, lines 73 and 74 - of single crystalline Si and thus allows an epitaxial growth of the crystal-forming material Si.

2.3 Since in the precharacterising part of Claim 1 the deposition surface area is required to have "a higher nucleation density than that of the adjacent deposition surface areas", such defined feature (a) is neither disclosed by the SiO_2 deposition surface area of the second embodiment of document D4 nor in document D5.

Figure 10 of document D5 only discloses explicit values of saturation densities of silicon nuclei on Si_3N_4 and SiO_2. There is no indication in document D5 to combine Si_3N_4 as "deposition surface area" with SiO_2 as "adjacent deposition surface area".

2.4 The deposition surface area disclosed in Figure 1 of document:

D6: Solid State Technology, volume 27, number 9, 1984, pages 239 to 243, cited by the Applicant during the examination proceedings,

involves epitaxial growth thereon without preceding creation of any nuclei.

2.5 Feature (b) concerning a limitation of the deposition surface area to such a size that only one single nucleus of supracritical size (remaining thereby stable) is formed within this area, is even not linguistically disclosed in document D4, in particular not in D4, column 2, lines 33 to 36, reading: "the diameters of the nucleation sites should be much smaller that the cross-sectional areas of the crystallites to be formed...". Feature (b) is also not derivable from document D3. According to the established legal practice of the EPO the technical disclosure of a document has to be interpreted as a whole. Thus, the limitation of nucleation to a single nucleus mentioned in paragraph 8 of document D3 concerns a topographically adjusted surface and not an area of different composition with higher nucleation density as claimed.

2.6 The deposition process claimed in feature (c) differs from that disclosed in document D4 by reacting the gaseous starting material not with hydrogen (D4, column 3, lines 26 to 30) but with a halogen oxidising

agent. Document D1 discloses feature (c) in a method for depositing amorphous silicon, which method does not comprise, in particular, features (a) and (b).

2.7 Document D7 (which forms prior art according to Article 54(3) EPC) discloses in column 15, lines 42 to 46 a deposition process wherein the starting materials SiH_2Cl_2, $SiCl_4$, $SiHCl_3$, SiF_4 and/or SiH_4 are reacted with HCl and/or H_2. Hence, document D7 discloses a reaction of the starting material with an oxidizing agent which is halogenic, i.e. which comprises halogen as a constituent. Present Claim 1 differs from the method according to document D7 in that the oxidizing agent is a halogen one, i.e. comprises only halogen atoms as constituents such as for example F_2, Cl_2, B_2, I_2 and ClF disclosed in the description of the present application, page 24, paragraph 1.

2.8 All other documents cited in the European Search Report or during the proceedings before the Examining Division, are less relevant than the documents discussed above.

2.9 Thus, in the Board's judgement the subject-matter of Claim 1 of the main request is novel in the sense of Article 54 EPC.

3. *Inventive step - Claim 1 - Main request*

3.1 In the second embodiment disclosed in document D4 at Figure 8 to 17 and column 3, lines 18 to 46, Si crystallites 16 are randomly oriented on SiO_2 surface 4 at nucleation sites which cannot be preselected; see column 3, lines 18 to 22. Hence, starting from this closest prior art, a first aspect of the objective problem underlying the present invention is to provide a method for producing one or more single crystals of a crystal forming material on a substrate which excludes

an epitaxial growth mechanism of the deposited material at desired sites; see also the description, page 20, lines 9 to 14. The above problem is solved by the combination of features (a) and (b) claimed in the precharacterising part of Claim 1.

3.2 Also having regard to the description, page 14, lines 1 to 14; page 19, line 25 to page 20, line 8; page 20, lines 15 to 27; and page 76, line 15 to page 77, line 7 a further aspect of the objective problem addressed by the present invention is the formation of a crystalline deposited film having uniform film quality and characteristics over a large area by using the deposition process according to feature (c) claimed in Claim 1. Feature (c) would clearly allow to keep the substrate temperature as low as 250°C (see the description of the present application, page 75 line 9) but does not exclude a higher substrate temperature. Since Claim 1 is not limited to a particular substrate temperature, the advantage of lower deposition temperatures cannot form part of the objective problem, see in particular paragraph II above.

3.3 In the assessment of an inventive step having regard to said first aspect of the objective problem according to paragraph 3.1 above, it has to be examined whether it was obvious to grow crystallites 16 in Figure 10 of document D4 at predetermined sites using measures (a) and (b) as claimed; i.e. whether it was obvious

(a) to localise non-epitaxial growth of single crystals at preselected desired sites on a surface by providing at the desired sites deposition surfaces having a higher nucleation density (in relation to the nucleation density on surfaces adjacent to the deposition surfaces) and

(b) to limit the size of the deposition surface for initiating the growth of one single crystal only i.e. to limit the size of surface area parts with higher nucleation density until -at chosen conditions of deposition - one stable nucleus forms on it and grows into one single crystal.

3.4 In the closest prior art according to the second embodiment of document D4 disclosed in Figures 8 to 13 and column 3, lines 18 to 46 the epitaxial crystal growth on the single crystal Si structure within the windows does not result in the desired crystals but controls the packing density of desired crystallites which grow randomly on an insulating layer within a desired surface area. An enlargement of the window area lowers the number of randomly distributed crystallites per unit area. This prior art does not allow one to recognise that on a surface needing nucleation for single crystal growth, such a nucleation can be initiated at desired sites by technical means. This embodiment gives a skilled person no hint to combine on one substrate two materials which exclude epitaxial growth and have different nucleation densities so as to increase nucleation density at the desired sites and to reduce the area of increased nucleation density (i.e. the deposition surface area) so as to initiate the growth of a nucleus of supracritical size (features (a) plus (b)).

3.5 There is no hint in document D4 to use Si_3N_4 for a deposition surface area, but only for the dielectric material embedding the crystallites 16 formed, see column 3, lines 47 to 54. In document D5 nucleation and growth of silicon films by chemical vapour deposition onto one and the same substrate surface is realised either on SiO_2 - substrates **or** on Si_3N_4 - substrates, but never on a substrate comprising both materials SiO_2 and

Si_3N_4 at the same time. The experimental results found separately for Si_3N_4 and SiO_2 as shown in Figure 10 of document D5 teach the skilled person only that the saturation density of silicon nuclei is higher on Si_3N_4 than on SiO_2. In the Board's view such experimental result does not suggest to a skilled person to provide both substances neighbouring to each other on the same substrate and to expose them simultaneously to the crystal forming material with a view to growing single crystals at predetermined nucleation sites.

3.6 Document D6, in particular Figure 1 with the corresponding description describes the conventional method of Epitaxial Lateral Overgrowth. In this method a window in layer with negligibly low nucleation density exposes a single crystal surface for the continued epitaxial growth of the single crystal. Thereby a skilled person is informed that single crystal growth at a preselected site is due to the existence of a single crystal lattice structure at that site. Hence, document D6 does not teach to deviate from epitaxial growth.

3.7 For the above reasons, feature (a) of the characterising part of the Claim 1 is held not to be obvious in view of the cited prior art.

3.8 As to the "first embodiment" in document D4 illustrated in Figures 1 to 7, column 2, lines 10 to 60, this describes literally the working principle of the present invention, i.e. to provide small areas of enlarged nucleation density for single crystal growth.

However, the Board regards it as decisive that there is insufficient information about the working concept of the claimed invention to make it obvious for a skilled person to carry out the claimed invention. The crucial

text at column 2, lines 33 to 35 reads: "Desirably, the diameters of the nucleation sites should be much smaller that the cross-sectional areas of the crystallites formed".

In the Board's view a skilled person sees in the above text only a desirable dimensioning of the deposition surface areas **relative** to the dimension of the final crystal. Hence from the text describing the first embodiment of document D4 a skilled person receives no information about the essential aspects of feature (b), i.e. that the deposition surface area **must** be so small that only "one single nucleus of supracritical size" is able to grow since otherwise no single crystal is formed. Providing the nucleation sites with their technically realisable minimum size which - following the Appellant's submission would be larger that claimed - would result in more than one stable nucleus being formed within the area of higher nucleation density, and finally the area would be covered by a polycrystalline film instead by a single crystal, so that the skilled person would be unaware of the concept of a differentiation of the nucleation density (i.e. selective nucleation) for growing single crystals.

3.9 The teaching in document D3, paragraph 8: "...it is necessary...to limit nucleation to a single nucleus and allow only this to grow" remains a desideratum of no technical help in relation to the objective problem. The intellectual support which a skilled person would derive from document D3 is restricted to the theoretical information that for single-crystal growth a single nucleus is needed. Document D3 is silent about the technical measures which are required for limiting nucleation in the formation of a single nucleus. There is no disclosure in document D3 (or in any other document) which logically relates the recommended

formation of one single nucleus with a corresponding **limited size** of a selectively nucleating **area** (i.e. an area of high nucleation density) on a free surface having elsewhere a negligible nucleation density.

3.10 For the reasons set out in paragraphs 3.8 and 3.9 above, feature (b) of the characterising part of Claim 1 is held not to be obvious in view of the cited prior art.

3.11 Having regard to the findings in paragraphs 3.7 and 3.10 above, the Board sees no reason to consider whether the further aspect of the present invention mentioned in paragraph 3.2 above contributes to the non-obviousness of the subject-matter claimed in Claim 1.

3.12 For the reasons set out above, in the Board's judgement the subject-matter of Claim 1 of the main request involves an inventive step in the sense of Article 56 EPC.

4. Thus Claim 1 of the main request is allowable under Article 52(1) EPC. Dependent Claims 2 to 19 concern particular embodiments of the method claimed in Claim 1 and are, therefore, also allowable.

Order

For these reasons it is decided that:

1. The decision of the Examining Division is set aside.

2. The case is remitted to the first instance with the order to grant a patent on the basis of the requested text (see paragraph V. above).

The Registrar: The Chairman:

M. Beer G. D. Paterson