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D E C I S I O N
of 17 March 1999

Case Number: T 0583/97 - 3.4.2

Application Number: 89303952.9

Publication Number: 0353837

IPC: H01M 4/52, H01M 4/32, H01M 10/30

Language of the proceedings: EN

Title of invention:
A nickel electrode for an alkaline battery

Patentee:
Yuasa Corporation

Opponents:
Société francaise SAFT
Japan Storage Battery Co., Ltd.

Headword:
-

Relevant legal provisions:
EPC Art. 100(b)

Keyword:
"Sufficiency of the disclosure (no)"

Decisions cited:
-

Catchword:
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Beschwerdekammern | Boards of Appeal | Chambres de recours

Case Number: T 0583/97 - 3.4.2

D E C I S I O N
of the Technical Board of Appeal 3.4.2
of 17 March 1999

Appellant: (Proprietor of the patent)
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Decision under appeal: **Decision of the Opposition Division of the European Patent Office posted 19 March 1997 revoking European patent No. 0 353 837 pursuant to Article 102(1) EPC.**

Composition of the Board:

Chairman: E. Turrini
Members: A. G. Klein
M. Lewenton

Summary of Facts and Submissions

I. European patent No. 0 353 837 (application No. 89 303 952.9) was revoked by decision of the Opposition Division, on the ground that its subject-matter lacked an inventive step within the meaning of Article 56 EPC.

The Opposition Division in its decision also ruled that the addition of ammonia, albeit not disclosed in the patent, certainly was an essential prerequisite to carry out the invention. Since the patent however mentioned that ammine complexes of nickel and magnesium or zinc should be produced first, for which the skilled person knew that ammonia was necessary, and since furthermore the necessity of adding ammonia could be derived from the discussion of the prior art in the patent, the Opposition Division did not endorse the opponents' objection under Article 100(b) EPC that the patent did not disclose the invention in a manner sufficiently clear and complete for it to be carried out by a person skilled in the art (see point 5 of the reasons).

II. The appellant (proprietor of the patent) lodged an appeal against the decision revoking the patent.

III. Oral proceedings were held on 17 March 1999, at which the appellant requested that the decision under appeal be set aside and that the patent be maintained on the basis of amended claims presented as his main, first auxiliary and second auxiliary requests with his letter dated 17 February 1999.

The respondents (opponents) requested that the appeal be dismissed.

Claims 1 and 2 of the appellant's main request read as follows:

"1. A nickel electrode including active material obtainable by the method of claim 2, comprising nickel hydroxide powder with zinc or magnesium in solid solution in crystals of said nickel hydroxide, the zinc or magnesium being present in the range of 3 to 10 wt% or 1 to 3 wt%, respectively, and the pore size within the powder being no greater than 3nm in radius and the total pore volume, when calculated from a desorption side of the nitrogen adsorption isotherm, being less than 0.05 cm^3/g.

2. A method of making active material for a nickel electrode, the active material comprising nickel hydroxide powder containing zinc or magnesium in solid solution in crystals of the nickel hydroxide, and the zinc or magnesium being present at from 3 to 10 wt% or 1 to 3 wt% respectively, the method comprising

adding ammonium sulphate to an aqueous solution of sulphates of nickel and the appropriate small amount of zinc or magnesium, forming ammine complex ions thereof, and

adding aqueous sodium or potassium hydroxide to bring the pH to a pH between 11 and 13 and deposit the nickel hydroxide crystals at said pH, by dropping the solution of complex ions into the aqueous hydroxide;

the pore size in the resulting powder being no greater than 3nm in radius and the total pore volume, when calculated from the desorption side of the

nitrogen adsorption isotherm, being less than 0.05 cm^3/g."

Claim 1 of the appellant's first auxiliary request reads as follows:

"1. A nickel electrode including active material comprising nickel hydroxide powder with zinc or magnesium in solid solution in crystals of said nickel hydroxide, the zinc or magnesium being present in the range of 3 to 10 wt% or 1 to 3 wt%, respectively, and the pore size within the powder being no greater than 3nm in radius and the total pore volume, when calculated from a desorption side of the nitrogen adsorption isotherm, being less than 0.05 cm^3/g,

said powder being obtainable by a method comprising:

adding ammonium sulphate to an aqueous solution of sulphates of nickel and the appropriate small amount of zinc or magnesium, forming ammine complex ions thereof, and

dropping the solution of complex ions into aqueous sodium or potassium hydroxide to deposit the nickel hydroxide crystals therein at a pH between 11 and 13."

Claims 1 and 2 of the appellant's second auxiliary request read as follows:

"1. A method of making active material for a nickel electrode, the active material comprising nickel hydroxide powder containing zinc or magnesium in solid solution in crystals of the nickel hydroxide, and the zinc or magnesium being present at from 3 to 10 wt% or 1 to 3 wt% respectively, the method comprising

adding ammonium sulphate to an aqueous solution of sulphates of nickel and the appropriate small amount of zinc or magnesium, forming ammine complex ions thereof, and

adding aqueous sodium or potassium hydroxide to bring the pH to a pH between 11 and 13 and deposit the nickel hydroxide crystals at said pH, by dropping the solution of complex ions into the aqueous hydroxide;

the pore size in the resulting powder being no greater than 3nm in radius and the total pore volume, when calculated from the desorption side of the nitrogen adsorption isotherm, being less than 0.05 cm^3/g.

2. A nickel electrode comprising a porous alkaline-proof metal substrate used as a current collector and a paste, which is loaded into the electrode, a principle component of the paste being active material attainable by the method of claim 1, wherein a small amount of cobalt exists in solid solution in the active material in addition to the zinc or magnesium by means of divalent cobalt compound, which forms cobalt complex ion when dissolved in alkaline aqueous solution, added to the active material powder in a range of 5 to 15 wt%, and wherein conductive additives are not included, the conductivity between the metal substrate and particles of the active material being substantially maintained only by virtue of the cobalt compound additive."

IV. The appellant's arguments in support of the admissibility of his requests, as far as they relate to the question of the sufficiency of the disclosure of the invention in the patent, can be summarised as

follows.

An active material for a nickel electrode in accordance with the present invention is produced in a two-stage process, which essentially comprises the forming of ammine complex ions of nickel and zinc or magnesium, followed by the decomposition of the complex ions by precipitation in a hydroxide solution to form a precipitate at a controlled pH of between 11 and 13. With respect to the first step, the patent explicitly discloses the adding of ammonium sulfate to a solution of nickel and zinc or magnesium sulfates, and it also expresses the functional target consisting in stabilizing ions of nickel and zinc or magnesium as ammine complex ions.

Ammine complex ions however are amongst the best known complexes, they have already been used in the manufacturing of nickel electrodes, as acknowledged in the introduction of the patent, and they can be easily identified by their characteristical blue colour. The skilled person would immediately recognise that the presence of ammonia is absolutely necessary to form these complex ions, and that such ammonia can be obtained by simply raising the pH of the acqueous solution into which ammonium sulfate has been added.

This is acknowledged in particular by the declarations by Prof. Kihara, Prof. Nagaosa and Dr Oshitani as filed with the letter dated 17 February 1999.

The quantities of the reagants are easy to determine, knowing in particular the coordination numbers for complexing. The concentration of the various salts in

the solution is not critical, and the admittedly huge amount of ammonium sulfate envisaged by Prof. Kihara would not impede the formation of the desired complex ions.

Anyway, the invention would still have worked, had the skilled person misunderstood the patent and directly added ammonia to the solution instead of raising its pH.

The appellant at the oral proceedings also demonstrated the formation of ammine complex ions of nickel by adding hydroxide solution to a sulfate solution formed by mixing 3M ammonium sulfate solution to 1M nickel sulfate solution. The procedure actually produced precipitation of nickel hydroxide, but the appellant indicated that this was accidental, and that the precipitate could anyway be easily removed from the solution before further processing. Thus, the skilled person using only common general knowledge would have had no difficulty to carry out the invention on the basis of the information given in the patent.

V. The respondents contested these submissions.

They stressed in particular that the explanations offered by the appellant at various stages of the procedure had undergone considerable evolution. The appellant in his response dated 7 December 1995 to the notice of opposition first suggested that the respondents in their experiments had wittingly selected non-optimal process parameters to demonstrate that the examples given in the patent did not work. In his letter dated 18 March 1996 the appellant then contended

that the respondents had not added sufficient ammonia to the sulfate solution. The appellant's interpretation of the patent to the effect that it would clearly teach the skilled person to provide alkaline conditions for the complexing step was not proposed until a late stage of the appeal procedure.

The respondents also submitted that the purely theoretical example given in the declaration by Prof. Kihara would in fact lead to a classical nickel hydroxide precipitate, as was evidenced by the declaration by Dr. Bernard filed with the letter dated 12 March 1999.

Reasons for the Decision

1. The appeal is admissible.

2. *Sufficiency of the disclosure*

2.1 The respondents at the oral proceedings expressed their doubts that the claims on file, and the amendments brought to them, actually met the requirements of Articles 84, 123(2) and 123(3) EPC, and they insisted that these issues should be examined and settled even before considering the question of the sufficiency of the disclosure.

In the Board's view, however, the sets of claims of the appellant's main, first and second auxiliary requests all comprise an independent product claim directed to a nickel electrode including active material comprising nickel hydroxide powder with zinc or magnesium in solid

solution in crystals of said nickel hydroxide, the zinc or magnesium being present in the range of 3 to 10 wt% or 1 to 3 wt%, respectively, and the pore size within the powder being no greater than 3nm in radius and the total pore volume, when calculated from a desorption side of the nitrogen adsorption isotherm, being less than 0.05 cm^3/g (see claim 1 of the main and first auxiliary requests and claim 2 of the second auxiliary request in conjunction with claim 1 to which it refers).

Thus, the question whether the disclosure of the invention in the patent is sufficiently clear and complete to allow a person skilled in the art to obtain such nickel electrode as defined in any of the above sets of claims is certainly a central issue in the assessment of the allowability of all of the appellant's requests, which can be examined first, independently of any formal objections which the respondents may wish to raise against further aspects of the claims.

2.2 The description of the patent discloses the manufacturing of the above nickel electrode by reference to two embodiments.

The first embodiment is described in column 4, line 57 to column 5, line 12:

"Ammonium sulfate is added to aqueous solution of nickel sulfate to which a small amount of zinc sulfate was added to stabilize ions of nickel and zinc ammine complex ions.

This solution is dropped into aqueous sodium hydroxide solution, while rapidly stirring it, to gradually decompose the complex ion and thus deposit and grow nickel hydroxide particles including the zinc in a solid solution state. This deposition is gradually performed in the weak alkaline solution of about pH 11 - 13 and at a temperature of about 40 - 50°C. Depending on the pH of the deposition solution, nickel hydroxide particles having various characteristics can be obtained."

The second embodiment, described column 9, lines 25 to 39, is distinguished from the first embodiment only by the use of magnesium instead of zinc.

The description does not offer any further details of the manufacturing processes.

The respondents submitted that the described processes neither achieved the formation of intermediate ammine complex ions, nor the deposition of a dense nickel hydroxide material, as was evidenced by test results produced with the notice of opposition dated 24 April 1995.

The appellant for his part admitted that the examples in the description do not actually result in the claimed material. The skilled person would however have easily recognised that the described processes can only achieve formation of ammine complex ions in the aqueous solution of ammonium, nickel and zinc or magnesium sulfates if the solution is rendered weakly alkaline. Achieving the claimed product did not require anything other than this obvious adaptation of the processes as

actually disclosed in the patent, the choice of the proper quantities and concentrations of the reagents not being critical by itself.

2.3 The Board in this respect however first observes that the description of the present patent only offers few details of the processes meant to achieve the claimed product, and that it lacks in particular any example of the required quantities and concentrations of the various reagents involved in the process. Such scant disclosure, in conjunction also with the long time needed for the deposition and growing of nickel hydroxide from the solution of sulfates as dropped into aqueous sodium hydroxide solution and the complexity of the measurements and calculations from a desorption side of the nitrogen adsorption isotherm as are necessary to ascertain whether the pore size and total pore volume of the powder obtained meet the limits set out in the claims, would already have imposed a significant burden on the skilled person striving to carry out the invention, even if the description had not failed to indicate how the ammine complex ions of nickel or zinc or magnesium were to be obtained. With respect to the time required for a single experiment, attention is drawn to the only example provided by the appellant in the opposition or appeal procedure of experimental conditions leading to the claimed material, as given in the paragraph "Experimental Conditions" of his letter dated 18 March 1996, according to which the dropping procedure was stopped after 24 hours and the slurry thus formed still had to be filtered, washed and dried.

In these circumstances, the skilled person would not in

the Board's opinion have easily recognised that his failure to produce the desired material was to be ascribed to any reason other than an incorrect selection of the process conditions. Incidentally, improper regulation of the process conditions was also the only explanation offered by the appellant in his response dated 7 December 1995 to the opponents' notice of opposition. The appellant admittedly later changed his argumentation, submitting instead that the process conditions were not critical, but he did not submit any evidence to this effect.

2.4 With respect to the appellant's argument that the skilled person on a proper interpretation of the description of the patent would have easily recognised the need for the aqueous solution of nickel and zinc or magnesium sulfates with added ammonium sulfate being rendered weakly alkaline, and that the use of such weakly alkaline solution would have immediately led to the desired material, the Board observes that the appellant's defence in the whole opposition procedure was actually based on the quite different argument that what was clearly needed to perform the described process was an addition of ammonia to the solution of sulfates. The only experimental data ever provided by the appellant, as given in his letter dated 18 March 1996, exclusively refer to the addition of a non-specified quantity of ammonium sulfate and aqueous ammonia to 2M nickel sulfate including 0.1 M zinc sulfate to produce the required nickel-ammine complex.

The appellant did not however produce any experimental data to establish that additional ammonia can be dispensed with altogether in case ammonium sulfate is

added to a weak alkaline solution of sulfates of nickel and zinc or magnesium. In this respect, the declarations by Prof. Kihara, Prof. Nagaosa and Dr Oshitani as filed with the appellant's letter dated 17 February 1999, which is about 10 years after the filing date of the patent and in full knowledge of the problem posed by the description and, possibly, of the latest developments of the invention, in broad terms merely describe procedures which they think the skilled person would have automatically followed but they do not in any way demonstrate that such procedures would also have actually resulted in the claimed product.

Concerning the declaration by Prof. Kihara, which is the only one to comprise quantitive details of a process supposed to achieve an ammine complex in an alkaline solution at a pH of about 10, the respondents pointed to an excessive amount of ammonium sulfate to be added to the solution, and, on the basis of a declaration by Dr Bernard filed with the letter dated 12 March 1999, they submitted that a substantial amount of classical nickel hydroxide would precipitate before complexing of the nickel and zinc ions present in the solution could occur. The appellant in this respect accepted that in the proposed process a substantial quantity of ammonium sulfate would simply be lost, but he contested that any premature precipitation of nickel hydroxide would substantially compete with the complexing reaction. The experiment conducted by the appellant in the oral proceedings however showed that, at least under uncontrolled conditions, such undesirable precipitation of nickel hydroxide could indeed occur even at a lower pH, which could in effect deter the skilled person from following the route

proposed by the appellant.

2.5 Finally, the Board also notices that the Opposition Division's conclusion that the invention was sufficiently disclosed in the patent was based on the assumption that the skilled person would proceed to the manufacturing of the claimed material by adding ammonia to the solution of ammonium, nickel and zinc or magnesium sulfates disclosed in the patent (see point 5 of the reasons). Such procedure would however in the Board's opinion not be consistent with the overall teaching of the present patent, which indeed presents the invention as a deviation from the prior art method in which ammine complex ions are stabilised from an ammonia aqueous solution (see the paragraph bridging columns 1 and 2 of the description). The addition of ammonia would not be consistent either with the necessity of providing ammonium sulfate as emphasised throughout the description, since ammonium sulfate would then be clearly superfluous.

The option of adding ammonia thus not being supported by the general teaching of the description, it cannot in the Board's view be validly relied upon to establish the sufficiency of the disclosure.

2.6 For these reasons, the European patent in the Board's opinion does not disclose the invention in a manner sufficiently clear and complete for it to be carried out by a person skilled in the art within the meaning of Article 100(b) EPC, which prejudices the maintenance of the European patent in any of the versions proposed by the appellant in accordance with his main, first auxiliary and second auxiliary requests.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

P. Martorana E. Turrini