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Anmeldenummer / Filing No / N^o de la demande : 79 300 554.7

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Bezeichnung der Erfindung: Thermoplastic compositions comprising blends of
Title of invention: styrene-acrylonitrile resin and nitrile rubber,
Titre de l'invention : and a process for their production

Klassifikation / Classification / Classement : C08L 9/02

ENTSCHEIDUNG / DECISION

vom / of / du 10 August 1990

Anmelder / Applicant / Demandeur :

Patentinhaber / Proprietor of the patent /

Titulaire du brevet : Monsanto Company

Einsprechender / Opponent / Opposant : Bayer AG

Stichwort / Headword / Référence : Resin-rubber blend/MONSANTO

EPO/EPC/CBE Article 54

Schlagwort / Keyword / Mot clé : "Novelty (affirmed - after amendment of claim)

Leitsatz / Headnote / Sommaire



Case Number : T 483/88

DECISION
of the Technical Board of Appeal 3.3.3
of 10 August 1990

Appellant : Monsanto Company
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Respondent : Bayer AG, Leverkusen
(Opponent) Konzernverwaltung RP
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Representative :

Decision under appeal : Decision of the Opposition Division of the European
Patent Office dated 21 July 1988 revoking
European patent No. 0 004 776 pursuant to
Article 102(1) EPC

Composition of the Board :

Chairman : F. Antony
Members : S. Schödel
J. Stephens-Ofner

Summary of Facts and Submissions

- I. European patent No. 4776 was granted with eleven claims in response to European patent application No. 79 300 554.7 filed on 4 April 1979.
- II. Notice of Opposition was filed by the Respondent (Opponent) requesting revocation of the patent in suit on the grounds of Article 100(a) and (b) EPC. Of the then cited documents only
 - (1) US-A-2 600 024 appeared to be relevant in this appeal.
- III. By decision dated 21 July 1988, the Opposition Division revoked the patent in suit for lack of novelty of its subject-matter. This decision was based on amended Claim 1, received on 26 October 1987, which was worded as follows:

"A composition comprising a blend of thermoplastic styrene-acrylonitrile (SAN) resin and nitrile rubber in which the rubber is present as a dispersion of particles in a matrix of SAN resin, and containing, per 100 total parts by weight of resin and rubber, 25 to 65 parts by weight of the SAN resin and 75 to 35 parts by weight of nitrile rubber, characterised in that the composition is thermoplastic, the rubber having been cured by simultaneously masticating and curing the rubber in a blend of rubber and molten resin using rubber curative in amounts and under time and temperature conditions known to give cured rubber products from static cures of the rubber in molds, said mastication being continued without interruption until the rubber of the blend has undergone gelation to the extent characteristic of rubber cured in molds and is at least 85% insoluble in toluene at room temperature."

In its decision, the Opposition Division held that (1) disclosed thermoplastic compositions comprising a blend of SAN resin and nitrile rubber within the claimed proportions in which the rubber was present as dispersion of small particles in a matrix of resin. The rubber was cured with a rubber curative and was at least 85% insoluble in toluene.

Although the feature "... the rubber having been cured .. in molds .." in the characterising portion of present Claim 1 did not appear in (1), it was clear from the statements in the description of the disputed patent that the same effect which was obtained by dynamic vulcanisation of a nitrile rubber/SAN resin blend could also be achieved by mixing the resin with a dynamically or statically vulcanised, powdered rubber. The compositions according to Claim 1 of the patent in suit did not offer any particularity over the known compositions and, consequently, did not relate to another, i.e. novel, invention within the terms of Article 54 EPC.

- IV. Notice of Appeal was lodged by the Appellant (Patentee) on 26 September 1988, the appeal fee being paid on the same day. A statement of grounds was submitted on 21 November 1988, together with a new set of seven claims. With letter of 10 July 1990, another set of six claims was delivered, the only amendment being that Claim 7 had been deleted from the aforementioned set of claims. Claim 1 has the following wording:

"A thermoplastic composition comprising a blend of thermoplastic styrene-acrylonitrile (SAN) resin and nitrile rubber in which the rubber is present as a dispersion of particles in a matrix of SAN resin, and containing, per 100 total parts by weight of resin and rubber, 25 to 40 parts by weight of SAN resin and 75 to 60 parts by weight of nitrile rubber, characterized in that the composition is elasto-plastic, said rubber is cured with a rubber curative

and at least 85% insoluble in toluene at room temperature and the rubber particles have a number average particle size of 50 μm or less."

Claims 2-5 are dependent on Claim 1 and Claim 6 is directed to a process for the production of compositions according to Claim 1.

V. In his written submissions, the Appellant argued essentially as follows:

- (a) Present Claims 1 to 6 were based on the claims of the patent as granted and should be formally allowable. The features newly introduced into Claim 1 could not be literally read from citation (1).
- (b) The compositions provided for by the patent in suit were elastoplastic. They could be processed and reprocessed without losing this property, although they comprised a vulcanised rubber. The preferred method to manufacturing them was dynamic vulcanisation.
- (c) The resin-rubber blends of the prior art were homogeneous intermixtures and not dispersions (cf. (1), column 6, line 25). The rubbery butadiene-acrylonitrile copolymer of (1) had in the unvulcanised state a methyl ethyl ketone (MEK)-insoluble gel content of from 40 to 100% obtained by hot-milling or by emulsion polymerisation in the presence of a small amount of a cross-linking agent. The said copolymer could still be vulcanised ((1) Examples 1, 2, 11).

- (d) For mixtures containing 25 to 50% b.w. of the hard resin, vulcanisation in the conventional manner was recommended (column 6, lines 53 to 63) which led to a thermoset material being not reprocessable thereafter, and not to an elastoplastic product which could be remoulded and allowed reutilisation of scrap material.
- (e) Nowhere in (1), in particular not in column 6, lines 30 to 42, was a rubber latex disclosed as would correspond to the definition provided for in Claim 1 on file. The cited section showed an intimate mixture of rubber and resin in a powder form, which could be shaped, but was not a thermoplastic elastomer.
- (f) The Respondent had picked out isolated portions of the disclosure of (1) and combined them out of their normal context. The subject-matter of the present invention was clearly novel over the prior art reference.

VI. In their counter-statement the Respondent contested the Appellant's arguments.

- (a) New Claim 1 was a mere combination of the Claims 1, 2 and 7 as patented and was still anticipated by (1).
- (b) The relative amounts of SAN resin/nitrile rubber of the compositions now claimed fell within the range of 25 to 50% for the SAN resin, which was disclosed in column 6, line 54 of (1) and which corresponded to 75 to 25% for the nitrile rubber.

- (c) From (1) it was clear that the rubber was present in the SAN matrix in the form of small particles (column 6, lines 30 to 42; column 1, lines 30 to 42; column 4, lines 18 to 22). This was elucidated by new document
- (9) Kunststoffe 57, 1967, 921, according to which, mechanical mixing of SAN resin and nitrile rubber ultimately gave a dispersion with small irregularly shaped rubber particles, the latter being in the order of magnitude of about 1 μm . There could be no doubt that the rubber particles in the dispersions resulting from (1) had a particle size of less than 50 μm .
- (d) Since the claimed compositions comprised at least 60% of vulcanised rubber, it was a matter of course that they were elastoplastic.
- (e) In (1) as well as in the patent in suit a clear distinction was made between the gel content of the rubbery ingredient, i.e. the portion insoluble in MEK or toluene, and the vulcanisates obtained therefrom by conventional vulcanisation.
- (f) New Claim 1 did not render the subject-matter of the patent in suit patentable. The same was true to process Claim 6, according to which the known compositions of Claim 1 were manufactured by merely applying the known method of dynamic vulcanisation.

VII. The Appellant requested that the decision under appeal be set aside and the patent in suit be maintained on the basis of Claims 1 to 6 of 10 July 1990.

By letter received on 8 February 1990, the Respondent submitted that he did not wish to take any further part in the appeal, and that he would therefore not comment on the Appellant's brief dated 21 August 1989. He nonetheless maintained his former request that the appeal be dismissed.

Reasons for the Decision

1. The appeal complies with Articles 106 to 108 and Rule 64 EPC and is, therefore, admissible.
2. The Appellant has filed fresh claims several times during opposition proceedings, again together with the appeal grounds, and once more on 10 July 1990. This is, in principle, an undesirable practice, and the Board has therefore considered whether the latest set of claims should be admitted at all. It has in this case decided to do so, because the said set differs from the preceding one only in the omission of Claim 7, a claim which the Board would anyway not have admitted because it had no counterpart in the granted claims (cf. T 295/87 of 6 December 1988, "Polyetherketones/ICI", to be published). In these circumstances, the latest set of claims served to render the claims in the appeal clearly allowable and was therefore to be admitted (cf. T 153/85, "Alternative Claims/AMOCO", OJ EPO 1988,1).
3. The claims before the Board meet the requirements of Article 123(2) and (3) EPC.

Claim 1 differs from the granted Claim 1 essentially in that the relative proportions of the SAN resin and the nitrile rubber in the compositions claimed are amended, that the rubber particles are defined by their number

average particle size and that the compositions are designated as elastoplastic. These amendments, in particular the numerical data inserted, find support in Claims 2 and 7 as originally filed and as granted. Dependent Claims 2 to 5 and process Claim 6, correspond to Claims 3 and 8 to 11 of the originally filed documents and of the published patent specification.

4. Citation (9) was - without appropriate justification - mentioned for the first time by the Respondent with his Statement of Grounds of Appeal and is, therefore, regarded as not submitted in due time within the meaning of Article 114(2) EPC. Having checked the relevance of (9), the Board considers it not relevant in the sense of influencing the outcome of this case and decides, in exercising its discretion conferred to it by Article 114 EPC, to disregard it.
5. The patent in suit is concerned with thermoplastic compositions comprising blends of thermoplastic styrene-acrylonitrile (SAN) resin and nitrile rubber exhibiting improved processing characteristics and physical properties and with a process for the production of such compositions.

The blends contain sufficient thermoplastic SAN resin to impart thermoplasticity and sufficient gelled nitrile rubber to impart rubberlike properties such as high extensibility and toughness. They are processable to products which form continuous sheets. The sheets can be reprocessed by melting and shaping.

6. The sole substantive issue to be decided in this appeal is the question of novelty of the subject-matter of the patent in suit vis-à-vis document (1).

6.1 The thermoplastic compositions of instant Claim 1 can be briefly characterised by the following features:

the blends comprise 25 to 40 p.b.w. of SAN resin and
75 to 60 p.b.w. of nitrile rubber;
the rubber component is cured with a rubber curative and has a toluene insoluble gel content of at least 85%;
the rubber particles have a number average particle size of 50 μm or less and are dispersed in a matrix of SAN resin;
the compositions are elastoplastic, i.e. they exhibit rubber-like elasticity yet are processable as a thermoplastic.

6.2 Document (1), on the other hand, describes composite thermoplastic homogeneous mixtures of a normally inelastic resinous thermoplastic SAN copolymer and a normally elastic rubbery butadiene-acrylonitrile copolymer, said thermoplastic resin comprising from 25 to 90% b.w. and said rubbery copolymer correspondingly comprising from 75 to 10% b.w. of the rubber-resin mixture (cf. Claim 3).

6.2.1 The rubbery copolymer of (1) is defined by its MEK-insoluble gel content, gel swelling index and Mooney viscosity. The MEK-insoluble gel content for the rubbery copolymer in the unvulcanised state should be from 40 to 100% b.w., with optimum results being achieved when the gel content ranges from 50 to 75%. If one of the three critical parameters specified for the rubbery copolymer falls outside the ranges indicated, the processing characteristics and the physical properties of the resulting blends with thermoplastic resins are impaired in one or more respects (column 3, paragraphs 2 and 4).

6.2.2 When determining the gel content of the rubber component by means of MEK or toluene it has to be observed that the two solvents exhibit entirely different solvent action upon the material in question. In the given example, a butadiene-acrylonitrile rubbery copolymer which was ground on the mill at 300°F (149°C) at the time when the toluene-insoluble gel level has reached 50% and 66% respectively, the MEK-insoluble level was only about 24% and 40% respectively (cf. column 2, paragraph 2). From these data it can be gathered that the toluene-insoluble gel level in general is higher than the MEK-gel level.

6.2.3 A closer study of the worked examples in (1) reveals that throughout 70/30 or 65/35 resin-rubber mixtures were investigated.

In Example 1, a commercial grade of an untreated and a hot-milled rubbery copolymer - the latter having a MEK-insoluble gel content of 65% - were compounded with sulphur and vulcanised for various times (30', 60' and 120' at 287°F) to determine the differences in physical properties of the untreated elastomer and the vulcanisate.

In Example 2, the aforementioned (unvulcanised) products were processed with SAN resin by banding the resin on a mill (at 300 to 320°F), then adding the rubber and blending thoroughly (10 to 12 minutes total milling time). The hot-milled rubber-resin mixtures exhibited improved physical properties over the untreated product. Further, it is mentioned that samples of rubber-resin mixtures prepared from the treated and the untreated rubber and containing a vulcanising system after vulcanisation in the conventional manner showed similar improvements in properties.

Rubbery copolymers, which were directly made by emulsion polymerisation, may contain a cross-linking monomer which is capable of joining molecule groups to form a cross-linked or gel structure. The cross-linking agent should be used in such a manner as to yield a product which is still rubber-like and can be handled by the usual rubber techniques. The amount of such cross-linking agent should, in general, range from 0.75 to 2% b.w. based on the weight of the other monomers present (cf. column 3, last paragraph, column 4, paragraph 1, column 11, lines 1-4). If the cross-linking agent, such as divinyl benzene, is added at the start of the emulsion polymerisation, the product obtained usually has from about 90 to 100% MEK-insoluble gel (cf. column 11, lines 18-23). Run G of Example 11, with a MEK-insoluble gel content of the hot-milled rubbery polymer of 89% and containing 1% divinyl benzene, was smooth in processing and gave, when blended with a resinous copolymer, satisfactory mixtures.

6.2.4 Specific mixtures containing from 25 up to 50% bw. of hard resin are disclosed in column 6, paragraph 5 of (1). These mixtures, after vulcanisation in the conventional manner, result in tough, flexible, leather-like materials. It is preferred to add vulcanising agents, such as sulphur, to the mixture and to vulcanise the resulting blends. The vulcanising agent should be used in such an amount as would vulcanise the rubbery copolymer to a soft vulcanised state if such rubbery copolymer were cured alone. Generally, from 0.5 to 5 parts of sulphur based on each 100 parts of total resin-rubber mix are employed.

6.3 When the claimed and the known systems are compared, it becomes evident that (1) does not expressly disclose at the places indicated SAN resin-nitrile rubber compositions having all the features specified in present Claim 1 of the patent in suit:

The claimed compositions are distinguished, in any case, from the known ones, referred to in Claim 3 of (1), in that they comprise a rubbery ingredient which is cured to the extent that its toluene-insoluble gel content is of at least 85% and which is present in the form of small dispersed particles (cf. section 6.2).

The compositions defined in the worked examples of (1) lie outside the scope of present Claim 1 and are, therefore, of minor relevance in deciding the issue of novelty; apart from this, in the samples with cured rubber copolymers, neither the gel content of the said rubber nor the particle size when blended with resin is reported (cf. section 6.2.3).

The statements in column 6, paragraph 5 of (1) imply that the balance to "the mixtures containing from 25 up to 50% by weight of the hard resin" would be 75 to 50% b.w. of rubber, which means that feed stock and amount of the claimed mixtures are in fact embraced by the known areas (cf. section 6.2.3). The resin-rubber mix is compounded with a vulcanising system and vulcanised in the conventional manner, i.e. under static conditions.

No further details are disclosed in (1), and in particular no instruction is available stating or implying that cross-linking of the rubbery copolymer has to be carried out to a point where it is 85% or more insoluble in toluene (or correspondingly in MEK) and that a dispersion of microsized, cross-linked rubber particles (having a number average particle size of 50 μm and less) has to be produced within the matrix of SAN resin, thus avoiding formation of thermoset, not reworkable material.

It is therefore at least questionable that the very general teaching of (1) - even if the additional information from the text of the description and the Examples quoted under

sections 6.2.1 to 6.2.4 are taken into account - would inevitably lead to the very specific compositions of present Claim 1 as alleged by the Respondent.

7. In a situation like this, where the parties make divergent submissions concerning the appearance of the claimed and known compositions, and the Board is in no position to establish the facts of its own motion, the burden of proof lies with the Respondent, who is the former Opponent. In the absence of any evidence, the Respondent's allegation that the blends of Claim 1 of the patent in suit are the same as those known from the prior art can only be held to be an unproven statement (cf. T 219/83, "Zeolites/BASF", OJ EPO 1986, 211).
8. For these reasons, document (1) is deemed not to be prejudicial as to novelty and the subject-matter of amended Claim 1 of the patent in suit is considered novel within the meaning of Article 54 EPC. The same applies *mutatis mutandis* to dependent Claims 2 to 5 and to process Claim 6. As a consequence, the decision of the Opposition Division can no longer be maintained.
9. The other requirements for patentability, in particular inventive step, have not yet been examined by the Opposition Division. The Board, therefore, deems it appropriate to remit the case to the first instance for further prosecution (Article 111(1) EPC).

Order

For these reasons, it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the first instance for further prosecution on the basis of Claims 1 to 6 of 10 July 1990.

The Registrar:

The Chairman:

M. Beer

F. Antony