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Datasheet for the decision of 27 November 2019

Case Number: T 2443/16 - 3.3.03

Application Number: 07767413.3

Publication Number: 2033990

IPC: C08L9/02, C08K5/098, C08K9/00, C09K3/10, F16G1/06

Language of the proceedings: EN

Title of invention:
NITRILE RUBBER COMPOSITION, CROSSLINKABLE NITRILE RUBBER COMPOSITION AND CROSSLINKED RUBBER

Patent Proprietor:
Zeon Corporation

Opponent:
ARLANXEO Deutschland GmbH

Relevant legal provisions:
EPC Art. 54, 56

Keyword:
Novelty- main request - (yes) - product-by-process feature makes structural contribution
Inventive step - main request - (yes)

Decisions cited:
T 0205/83

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Case Number: T 2443/16 - 3.3.03

D E C I S I O N
of Technical Board of Appeal 3.3.03
of 27 November 2019

Appellant: (Opponent)	ARLANXEO Deutschland GmbH Alte Heerstrasse 2 41540 Dormagen (DE)
Representative:	Rupp, Christian Mitscherlich PartmbB Patent- und Rechtsanwälte Sonnenstraße 33 80331 München (DE)
Respondent: (Patent Proprietor)	Zeon Corporation 6-2, Marunouchi 1-chome Chiyoda-ku Tokyo 100-8246 (JP)
Representative:	Adam, Holger Kraus & Weisert Patentanwälte PartGmbB Thomas-Wimmer-Ring 15 80539 München (DE)
Decision under appeal:	**Interlocutory decision of the Opposition Division of the European Patent Office posted on 22 July 2016 concerning maintenance of the European Patent No. 2033990 in amended form.**

Composition of the Board:

Chairman	D. Semino
Members:	M. C. Gordon
	R. Cramer

Summary of Facts and Submissions

I. The appeal lies from the interlocutory decision of the opposition division posted on 22 July 2016 according to which European patent number 2 033 990 could be maintained in amended form on the basis of the main request, filed with letter of 21 October 2014 and an amended description filed during the oral proceedings on 29 June 2016.

II. The patent as granted had 13 claims whereby claims 1 and 13 read as follows:

"1. A nitrile rubber composition comprising a nitrile rubber (a) including 10 to 60 wt% of an α,β-ethylenically unsaturated nitrile monomer unit and 40 to 80 wt% of a diene monomer unit and/or α-olefin monomer unit, and having iodine value of 100 or less, an α,β-ethylenically unsaturated carboxylic acid metallic salt (b), and silane-treated fumed silica and/or calcinated wet silica (c).

13. The nitrile rubber composition as set forth in any one of claims 1 to 7, wherein said silane-treated fumed silica is obtained by surface treatment of fumed silica with a silane compound before forming the nitrile rubber composition."

III. An opposition against the patent was filed invoking the grounds pursuant to Article 100(a) EPC (lack of novelty, lack of inventive step) and Article 100(c) EPC.

The following documents, *inter alia* were relied on by the opponent:

Filed with the notice of opposition:

D1: US-A-5 208 294
D3: US-A-4 918 144

Filed subsequently by the opponent:

D6: EP-A-952 185
D7: EP-A-753 549

Submitted by the patent proprietor:

D9: experimental report.

Claim 1 of the main request differed from granted claim 1 in that the additional feature of granted claim 13 was added at the end of the claim.

IV. According to the decision, D6 and D7 were admitted to the proceedings. D9 was not admitted to the proceedings.

The claims of the main request met the requirements of Article 123(2) EPC.

The subject-matter claimed was furthermore held to be novel with respect to D1 since it would be clear that the specified pretreatment of silica with silane resulted in a higher yield of silane on the surface of the silica. Furthermore in the case of *in situ* addition, i.e. combination of the rubber, silica and silane in a single step, there would be migration and some reaction of the silane with the rubber initiated by the vulcanisation agents. Thus the manner of incorporating the silane necessarily had an influence on the structure of the product.

The subject-matter was held to meet the requirements of inventive step, this conclusion being reached on the basis of D3 as representing the closest state of the art (D1 being considered unsuitable), the decision further relying on D6 and D7.

V. The opponent (appellant) filed an appeal against the decision.

Together with the statement of grounds of appeal

D10: Experimental report

was submitted, which was stated to be in reaction to the submissions of the patent proprietor at the oral proceedings before the opposition division.

Objections of lack of novelty with respect to D1 and lack of inventive step with respect to either of D1 or D3 as closest prior art were maintained.

VI. Together with the reply the patent proprietor (respondent) defended the patent in the form as upheld. Furthermore auxiliary requests 1-7 from the opposition proceedings (filed with letter of 21 October 2014) were stated to be maintained, but not resubmitted.

A further document:

D11: "A Guide to Silane Solutions - Silane Coupling Agents" (company brochure of Xiameter, 2009)

was cited.

Submissions were made regarding admittance of D9 to the

proceedings.

Regarding experimental report D10 filed by the appellant, the document was dealt with in substance. An objection to admittance thereof was not raised.

VII. The Board issued a summons to oral proceedings and a communication.

VIII. With letter of 20 November 2019 the respondent filed a further set of claims as auxiliary request 8.

IX. Oral proceedings were held before the Board on 27 November 2019.

X. The arguments of the appellant in respect of the main request can be summarised as follows:

(a) Novelty

Example 5 of D1 disclosed a composition of nitrile rubber, silica and a silane which were blended together in a single step. The product by process feature of operative claim 1 relating to prior treatment of the silica with the silane agent could not confer novelty. It was inevitable that in the process of D1 there would be an interaction between the silica and the silane meaning that silica treated with silane would be present.

Experimental report D10 confirmed that the properties of the compositions in terms of tensile strength, elongation and heat ageing were substantially identical regardless of how the silica and silane were introduced. Thus, since the product-by-process feature did not result in any

difference in the properties of the product, it could not be relied upon to confer novelty.

The proposed differences in structure of the silica/silane components on the basis of D11, referred to by the respondent, were not decisive for the question of novelty. At most this document explained how the silica and silane might be differently distributed within the mass, but this still indicated that silica treated with silane would be present in the case of *in situ* addition and furthermore did not provide any evidence in respect of a difference in properties of the obtained rubber composition. In particular the claim did not have any restriction in terms of the degree of surface treatment of the silica.

On this basis the composition of claim 1 was not novel with respect to example 5 of D1.

(b) Inventive step

D3 constituted the closest prior art, although D1 could also be taken as an alternative starting point.

With respect to D3 the distinguishing feature was the nature of the silica. The citation employed wet silica whereas the claim required either calcined wet silica or silica surface treated with silane.

The available evidence did not demonstrate a technical effect. In particular the silicas employed in the examples and comparative examples differed in a number of features, such as particle size and pH meaning that no effect could be

ascribed exclusively to the distinguishing feature. Thus the objective problem to be solved was the provision of further compositions. D6 or D7 taught that this aim could be achieved by pretreatment of the silica.

Starting from D1 the distinguishing feature would be the pretreatment of silica with silane rather than *in situ* treatment. No effect resulted from this difference meaning that the further considerations on obviousness were the same as when starting from D3.

XI. The arguments of the respondent in respect of the main request can be summarised as follows:

(a) Novelty

The evidence of the appellant (D10) failed to show that in the *in situ* blending method of D1, example 5, silane treated silica as required by operative claim 1 would arise. Furthermore D10 only reported a selection of the physical properties and hence could not conclusively demonstrate the nature of the products.

Considering that in the *in situ* method there would be the possibility of reaction between the free silica, free silane and the rubber, it was inevitable that different products would result than in the case where preformed silane treated silica was employed. This was confirmed by D11 and in particular by D9 which showed differences in the properties of the resulting products, in particular heat ageing resistance, depending on the two methods of mixing. This established that the

product by process feature conferred structural features to the product and hence could be relied upon to establish novelty.

On this basis novelty of the composition of claim 1 over example 5 of D1 should be acknowledged.

(b) Inventive step

Closest prior art was D3. D1 was more remote and did not address the same problem as the patent in suit so that it could not be considered as an alternative starting point.

D3 considered many possible fillers. Silica was not emphasised and was not preferred but was employed only in one - not particularly good - example, namely run 26 which employed wet silica. Thus the claimed subject-matter differed therefrom in that calcined wet silica or silane treated fumed silica were specified. The examples of the patent demonstrated for both types of silica (calcined wet silica and surface treated fumed silica) good properties which were barely worsened by heat ageing. In contrast as shown by the data in the patent in suit and those in D9, the comparative compositions (*in* situ addition of silane and precipitated wet silica rather than calcined wet silica) demonstrated considerable deterioration following heat ageing in respect of both embodiments. D10 confirmed this result for the case of pretreatment of the silica, even if the extent of improvement/degree of retention was not as great as in the examples of the patent and D9.

Although the silicas employed in the examples and

comparative examples differed in terms of particle size/surface area, D9 established that this had minimal to no effect on the properties of the compositions. In contrast the nature of the treatment of the silica was shown to have significant effects, outweighing any arising from particle size. Regarding the objection of the appellant relating to differences in pH between the silicas employed in the examples and comparative examples of the patent, particularly in the case of calcined wet silica, this was an inevitable consequence of the different surface treatments and thus was directly linked to and a consequence of the distinguishing feature.

Thus the objective problem was to provide compositions with improved properties.

The solution - employment of the particular types of silica - was not rendered obvious by the prior art.

XII. The appellant requested that the decision under appeal be set aside and that the patent be revoked.

XIII. The respondent requested that the appeal be dismissed, or alternatively that the decision under appeal be set aside and the patent be maintained on the basis of one of the sets of claims according to auxiliary requests 1 to 7 filed with the letter of 21 October 2014, or according to auxiliary request 8 filed with the letter of 20 November 2019.

Reasons for the Decision

1. Main request

1.1 Admittance of documents D9, filed by the respondent during the opposition proceedings and D10 (appellant) and D11 (respondent), filed during the appeal proceedings

No objections were raised to the admittance of these documents by the parties and the Board is aware of no reason to take a different position. Documents D9 to D11 are therefore in the proceedings.

1.2 Novelty

Claim 1 specifies that the silane treated fumed silica is obtained by surface treatment of fumed silica with a silane compound prior to forming the nitrile rubber composition.

D1 discloses in example 5 the preparation of a composition of hydrogenated nitrile rubber, an unsaturated carboxylic metal salt (zinc diacrylate) as required in operative claim 1, silica and a silane.

The last two components are combined together with the rest of the components in the same step.

The only question under dispute is whether the product by process feature i.e. the requirement that the silica be surface-treated with a silane compound before forming the nitrile rubber compositions influences the properties of the obtained products, i.e. makes a structural contribution thereto, so as to result in a different product (with reference to T 205/83, OJ EPO

1985, 363, section 3.2.1 of the reasons; see also "Case Law of the Boards of Appeal of the European Patent Office", 9th Edition section II.A.7.2).

Evidence has been advanced in the form of D9 by the respondent, example 3 and comparative example 7 being relevant:

Table 1

			Examples			Comparative Examples						
			1	2	3	1	2	3	4	5	6	7
Components (parts)	Zinc dimethacrylate		15	15	15	15	15	15	—	15	15	15
	Silica (1) "Aerosil R972"		40	—	—	—	—	—	40	-	-	-
	Silica (2) "Carplex CS-7"		—	40	-	—	—	—	—	—	-	-
	Silica (3) "Nipsil VN-3"		—	—	—	40	40	—	—	—	-	-
	Silica (4) "Aerosil 200"		—	—	—	—	—	40	—	40	-	-
	Silica (5) "AerosilRNX 90G"		—	—	40.4	—	—	—	—	—	-	-
	Silica (6) "Aerosil®90G"		—	—	-	—	—	—	—	—	40	40
	Silane Coupling Agent (1) "A-172"		—	—	—	—	2	—	—	—	-	-
	Silane Coupling Agent (2) "KBM-503"		—	—	—	—	—	—	—	2	-	-
	Silane Coupling Agent (3) HMDS		—	—	—	—	—	—	—	—	—	0.91
Normal Physical Properties	Tensile Strength	(MPa)	26.5	25.5	26.7	25.7	27.0	25.3	24.1	25.8	26.2	27.1
	Elongation	(%)	470	430	450	480	460	460	430	430	460	440
	100% Tensile Stress	(MPa)	7.3	8.5	7.7	6.8	6.8	7.3	3.2	7.5	7.1	7.8
Heat Aging Test	Tensile Strength	(MPa)	27.6	27.6	27.1	28.0	27.2	26.6	25.0	25.7	27.9	27.5
	Elongation	(%)	390	350	380	270	260	290	360	300	290	280
	Rate of Change in Elongation	(%)	-17	-19	-16	-44	-43	-37	-16	-30	-37	-36

and by the appellant with D10, example 1 and 4 (comparative) being significant:

Tabelle 2: **Ergebnisse**

Compound		1	2	3	4	5
Normal Physical Properties						
elongation	%	464	310	296	418	363
tensile strength	MPa	32,4	32,4	31,3	35,6	36,1
Heat Ageing Test (150°C, 24h)						
elongation	%	430	276	265	388	337
tensile strength	MPa	33,5	31,3	30	36,6	37,3
change of elongation	%	-7	-11	-10	-7	-7

In each case the first named examples employ pre-compounding as required by the product-by-process feature of claim 1 and the other named examples employ the *in situ* blending method of D1.

In both cases differences associated with the manner in which the silica/silane component was generated are observed in terms of absolute values of tensile

strength (D9 and D10). D9 also shows that the retention of these physical properties after ageing is improved as a result of the nature of the treatment.

Thus the evidence is that the manner of treatment influences the properties of the final product.

This quantitative observation is consistent with the qualitative arguments put forward by the respondent with reference to D11, demonstrating differences in the structure and distribution of silica and silane depending on the manner of introduction (page 5 of D11 and arguments on page 6 of the reply to the statement of grounds of appeal).

In the light of the evidence and the arguments advanced, the Board is satisfied that the product-by-process feature results in a qualitative difference in the structure of the filler, which is manifested by the properties of the final composition.

The only conclusion that can be reached is that the manner of incorporation of the silica and the silane - even if the exact nature of the structure at a molecular level is not known, or at least is a matter of dispute between the parties - is convincingly shown to exert an effect on the overall properties of the compositions, i.e. to result in a structural effect which can be measured and thus result in a different product (cf T 205/83, *infra*).

Under these circumstances it has to be concluded that the product-by-process feature results in a difference in the obtained products, and thus confers novelty.

It is correct, as argued by the appellant, that the

claim contains no requirement in respect of the extent of surface treatment and is of an open formulation thus permitting the presence of other types of additives. However the evidence provided suggests that there will be a structural contribution even if only a portion of the silica has been so treated. Furthermore the appellant has not advanced any arguments or evidence to demonstrate that in a "hybrid" system either having a mixture of treated and non-treated silica, or a system prepared by incorporating a portion of pretreated silica, a portion of untreated silica and a portion of treating agent there would be a different outcome, which would invalidate the evidence showing that incorporating the pretreated silica makes a structural contribution to the composition. Accordingly taking this aspect into consideration does not lead to a reason to modify the above assessment.

Regarding the second embodiment, calcined wet silica, no objections of lack of novelty were raised and the Board is aware of no reason to take a diverging position.

1.3 Inventive step

1.3.1 Closest prior art

The patent in suit is directed to the provision of rubber compositions having excellent tensile stress and heat resistance (paragraph [0001]). The envisaged uses are in particular as components in the engine compartments of automobiles e.g. hoses, gaskets, which articles are subjected to severe heat stress during use (paragraph [0002]).

By common consent the closest prior art was considered

to be D3, although the appellant submitted that also D1 could be considered.

D3 relates to the provision of vulcanizates having excellent strength properties (column 1, lines 4-8, 19-28). According to column 3, lines 48-51 the compositions have higher tensile strength, lower dynamic heat generation and better heat ageing resistance than prior art compositions.

D1, which in the alternative was proposed by the appellant as representing the closest prior art relates to hydrogenated nitrile rubber based vulcanizates having improved load bearing properties, improved abrasion resistance, reduced heat build up (abstract, first paragraph of description, section "Summary of the invention" in column 1). Heat resistance is discussed in column 3, lines 3-12 in respect of products of the prior art. The compositions are useful in belt drives, rubber covered rolls and high pressure hoses. The specific technical requirements and end applications as set out in the patent in suit and D3 are not addressed in D1.

The problem addressed by D3, i.e. improvement in tensile strength, and good heat resistance (ageing) is much more closely aligned with the problem set out in the patent in suit than that addressed by D1.

Accordingly the Board concludes that D3 represents the closest state of the art while D1 is a much more distant document which the skilled person faced with the object of the patent in suit would not have considered as the starting point.

1.3.2 Distinguishing feature

According to the opposition division (decision section 2.3, 3rd paragraph), the appellant (statement of grounds of appeal section V.2, 4th paragraph), and the respondent (rejoinder to the statement of grounds of appeal, section 3.4.2, first paragraph), the sole difference with respect to the relevant disclosure of D3 is the nature of the silica. Indeed D3, run 26 employs Carplex #1120 as wet silica, whereas the claims require silane surface-treated silica or calcined wet silica.

The Board can also concur with this conclusion.

1.3.3 Technical effect

(a) Embodiment surface-pretreatment of silica with silane

Experimental report D9 provides comparisons of the different modes of treating silica. The results relevant for this decision were summarised in a table provided by the respondent with letter of 20 November 2019 (page 3).

			Examples		Comparative Examples		
			1	3	3	6	7
Normal Physical Properties	Tensile Strength	(MPa)	26.5	26.7	25.3	26.2	27.1
	Elongation	(%)	470	450	460	460	440
	100% Tensile Stress	(MPa)	7.3	7.7	7.3	7.1	7.8
Heat Aging Test	Tensile Strength	(MPa)	27.6	27.1	26.6	27.9	27.5
	Elongation	(%)	390	380	290	290	280
	Rate of Change in Elongation	(%)	-17	-16	-37	-37	-36
Aerosil			R972	NX90G	200	90G	90G*

*in situ silane treatment

The details of the silicas employed is as follows: R972: silane treated fumed silica with specific surface area of 100 m^2/g obtained by treating

Aerosil 130 with dimethyldichlorosilane (patent in suit, paragraph [0075], last three lines "Note 1");
NX90G: silica having specific surface area of 65 +/- 15 m^2/g treated with hexamethyldichlorosilane ("HMDS" - D9, page 1, 1st paragraph, page 2, first paragraph);
200: fumed silica having specific area of 200 m^2/g (patent in suit paragraph [0079] last two lines);
90G: untreated silica having specific surface area 90 +/- 15 m^2/g (D9, page 1, second paragraph);
90G*: silica 90G with *in situ* HMDS treatment - D9 section A "Comparative Example 7".

From these data the following is apparent:
Considering the three examples based on silica 90G with various types of treatment (example 3, comparative example 7) and without treatment (comparative example 6) it is seen that pretreatment leads to improvements in elongation retention after heat ageing (example 3 and comparative example 7). This improvement is seen both with respect to the non-treated silica (comparative example 6) and that having *in situ* treatment (comparative example 7), whereby the results of the *in situ* treatment are largely identical to those where no silane treatment of any kind is employed (comparative examples 6 and 7).

The trend is also consistent between the two "inventive" examples and the three comparative examples shown in the above excerpt of the data of D9, differences in particle size of the silicas notwithstanding.

Regarding the particle size these data do not

support the position of the appellant that the differences in particle size of the silica employed in the various examples invalidates the conclusions or results thereof, but that the overriding influence on the outcome is the method of pretreatment. This lack of influence of the particle size on the observed effect is also apparent from the broader set of data contained in D9.

This evidence shows that for the embodiment relating to pretreatment of silica with silane the effect is to improve the retention of physical properties (in particular elongation) after heat ageing.

(b) Embodiment calcined wet silica

In the patent example 2 employs "Carplex CS-7" which according to "Note 2" in paragraph [0076] is calcinated wet silica having specific surface area of 133 m^2/g and pH 6.9. Comparative example 1 employs instead "Nipsil VN-3" which according to "note 3" in paragraph [0077] is precipitated wet silica having specific surface area of 195 m^2/g. The pH is not reported.

The data in table 1 of the patent show that in this case as well there is an improvement in retention of elongation following heat ageing when employing the silica according to the claims.

It is correct, as argued by the appellant that the pHs of the two silicas are not shown to be the same (see section IX.(b), 3rd paragraph, above). However it is credible, as submitted by the respondent

(above, section X.(b), final section) that the difference in pH of the inventive and comparative silicas was a direct consequence of, and consequently a reflection of, the nature of the treatment, i.e. the calcination and hence was related to the distinguishing feature. Accordingly any such discrepancy in this respect does not invalidate the results or conclusions drawn on the basis thereof.

The conclusion is thus that the nature of the treatment of the silica (calcination in this case) gives rise to the same technical effect as observed for the pretreatment of silica with silane, namely an improvement in particular in elongation properties following heat ageing.

1.3.4 Objective technical problem

In the light of the foregoing, the objective technical problem in the case of both embodiments can be formulated as the provision of nitrile rubber compositions exhibiting better resistance to heat ageing.

1.3.5 Obviousness

Closest prior art D3 itself contains no recognition of any influence of the nature of the treatment of silica on the resistance of the compositions to heat ageing.

D6 is directed to the provision of compositions for tyres and tyre treads (paragraph [0001]) based on rubber compositions containing silane treated inorganic fillers. Paragraph [0005] reports that the rubber compositions containing silica fillers subjected to a

hydrophobic treatment can be processed without leading to loss of filler into drainage water, and results in improvements in the processing properties of the rubber composition, in particular longer processability times and lower viscosity. The obtained compositions have improved shock absorbing properties and abrasion resistance. There is no recognition herein that there is any benefit in terms of heat ageing.

The patent in suit is in contrast directed to a different area of end use, namely automotive components such as belts, gaskets, hoses, packings, oil seals, which components are exposed to heat and hence are required to exhibit heat resistance (paragraph [0002]) as expressed in the formulation of the technical problem.

Accordingly there is no guidance in D6 to address the objective technical problem as set out above. Furthermore it is observed that there is no particular emphasis in D6 on silica in any form, this being merely one of 7 different types of inorganic filler considered (paragraphs [0029] and [0030]}.

Similarly D7 relates to the provision of materials for tyres and to rubber compositions containing surface modified silica based fillers (page 1, lines 1-19). The specific suite of technical requirements and problems as addressed by the patent in suit is not discussed in D7. Consequently there is no indication in D7 that any improvement in ageing resistance would arise as a result of the specific nature of the surface treatment of the silica.

Accordingly neither the closest prior art D3 on its own, nor in combination with either of the other

documents invoked - D6, D7 - provides a teaching which would lead to the claimed subject-matter as a solution to the objective problem.

On this basis the presence of an inventive step has to be acknowledged.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

A. Pinna D. Semino

Decision electronically authenticated