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(54) Title: POLYAMIDE-BASED SYNTHETIC SAUSAGE CASING ABLE TO BE FILLED WITHOUT STRETCHING AND METHOD OF PRODUCTION THEREOF

(57) Abstract: The invention relates to single- or multilayer synthetic heat-shrinkable sausage casings that one can fill without preliminary humidification and virtually without stretching and make as a result taut sausages, and also relates to a method of production of such casings. At least one polyamide layer of the casing is made of polyamide resin based on at least one aliphatic (co)polyamide, selected from polyamide-6, polyamide-66 or copolyamide-6/66. The casing, being ready to be filled, has following characteristics: the Young's modulus is not less than 300 MPa in the machine direction and not less than 400 MPa in the transverse direction; on exposure to an air with relative humidity 60% and a temperature of 25°C for 5 days its shrinkage value is no more than 2% both in the machine and transverse directions; on exposure to a water with temperature 40°C for 2 hours its shrinkage value is 3-10% in the machine direction and 4-10% in the transverse direction; on exposure to water with temperature 80°C for 30 minutes its shrinkage value is 8-15% in the machine direction and 9-18% in the transverse direction. The method of production of such sausage comprises extrusion of single- or multilayer extrudate, resulting in tubular film, orientation drawing and thermofixation of the tubular film, and the subsequent additional treatment. The subsequent treatment consists in additional drawing of the oriented thermofixed tubular film, having temperature of 60-140°C, by 3-8% in the machine direction and 4-10% in the transverse direction; subsequent cooling of this tubular film, while it is in the drawn (strained) state, to the temperature that is not higher than glass transition temperature of the polyamide composition of the polyamide layer; and its winding up on a core. Said steps of the process can be performed at the aggregate that is either built in the line for accomplishment of the previous steps of process, or at a separate machine.



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Polyamide-based synthetic sausage casing able to be filled without stretching and method of production thereof

### Field of the Invention

5 The invention relates to single- or multilayer synthetic heat-shrinkable sausage casings that one can fill without preliminary humidification and virtually without stretching and make as a result taut package, for instance sausages. Additionally the invention relates to a method for production of such casings.

### Background of the Invention

10 Synthetic casings for sausage products such as sausages, frankfurters, hams, liver pastes enclosed in a casing and so on are well-known from the prior art. Such film casings are practical in use and allow making packed products attractive for consumers. Particularly, such casings allow making taut sausages without wrinkles. Such taut sausages do not only have attractive appearance but also guarantee an absence of liquid,  
15 fat or gel between the casing and sausage meat (so called "purge" or "cook out").

There are a few known approaches for making the taut sausages. The first of them is based on using of phenomenon of heat shrinkability in making of sausages in plastic casings. It consists in filling of heat-shrinkable plastic casing without significant expanding with sausage meat and subsequent cooking or scalding of the sausages. Sausage  
20 meat expands during the cooking and stretches the casing. At the beginning of subsequent cooling (that begins from the surface of a sausage) the new geometry of the sausage casing is fixed because it gains low temperature while sausage meat remains still hot and expanded. Therefore after complete cooling of the sausage when sausage meat decreases in its volume the sausage casing covers it not-tautly and even can form wrinkles. Taut  
25 sausages are made by their short-time heating usually by means of treatment with hot water or steam, which results in thermal shrinkage of the casing. Such the casings (single- or multilayer) can be made of various resins such as polyvinylidene chloride (hereinafter referred to as PVDC), polyolefins: polypropylenes (PP) and polyethylenes (PE) of various grades and types, polyesters such as polyethylene terephthalate, polybutylene terephthalate  
30 and others, and also polyamides. Heat shrinkable casings are made by methods of orientation drawing with subsequent non-complete or no thermofixation (or annealing). Made thereby casings are characterized with unstable crystalline phase comprising a lot of small and imperfect crystals that can be destroyed at relatively low temperatures. Their destruction (actually melting) results in formation of additional amorphous phase, which

due to thermal motion of polymer chains, trends to cease to be oriented, starting a development of shrink forces. When such processes take place during sausage cooking, the sausage meat expanding does not allow this forces relaxation and after the cooling a new crystalline phase, which fixes new geometry of casing, is formed. After the short-time heating the crystals destroy again but in this case nothing prevents the relaxation of shrink forces and casing shrinkage takes place. It is obvious that such approach which implies an additional heat treatment of sausages after their cooling requires undesirable excess power and labor inputs.

European patent application № 0 974 452 (published 2000.01.26) disclosed a stretched (drawn) multilayer film casing having desirable heat-shrinkability level and decreased heat-shrinkage stress value. The casing comprised a pair of outer layers comprising polyolefin resins of identical species, and a gas-barrier intermediate layer comprising a polyamide resin. The film casing was made according to a usual process for oriented films with thermofixation step carried out at a low temperature by hot (60-98°C) water or steam. Resulted casing had heat-shrinkage stress maximum of 2 MPa at 50°C and hot-water shrinkability at 90 °C of 5 - 20% in each of machine (longitudinal) direction and transverse direction. Such casing can be filled with sausage meat automatically or manually. Resulted sausage was cooked according to a usual cooking process, cooled and then put into hot water. In the inventor's opinion, limited heat-shrinkage stress was needed to avoid violation of packing integrity (rupture of casing or its sealed seams, etc.) during cooking of sausages.

Second approach is based on elastic properties of plastic casings. In this case a range of suitable materials is shorter. It is possible to use such resins as thermoplastic polyurethanes for their production, or a less known spatiality block-copolymers characterized with elastic properties (thermoplastic elastomers). As a rule such materials are very expensive and difficult in processing. Therefore the most widespread raw materials for making elastic sausage casings are aliphatic polyamides. They are used both as a basic raw material of single-layer casings and as a raw material for one of layers (usually thickest) in multilayer casings. Such casings commonly have good barrier properties towards moisture and oxygen, what gives a possibility to store food products packed therein for quite long time.

Commercial aliphatic polyamides such as polycaprolactam (hereinafter referred to as polyamide 6 or PA 6), polyhexamethyleneadipamide (hereinafter referred to as polyamide 66 or PA 66) and other aliphatic polyamides in non-oriented state (for instance,

in a form of cast films) have no elasticity and, on the contrary, they tend to undergo irreversible deformations. The following process is used to provide them with elastic properties: extrusion of a cast tubular film (or primary tube) from single- or multilayer extrudate; orientation drawing of the cast tubular film resulting in secondary tube; most possible complete thermofixation of the secondary tube resulting in ready-to-use sausage casing.

The same process was disclosed, for instance, in a US Pat. № 4 303 711 (published 1981.12.01). It results in casings which have a capability of great reversible deformations (high elasticity) and also remarkable mechanical strength and dimensional stability. Relative dimensional stability is typical for these casings even at elevated temperature and high humidity (while it is immersed into hot water at 80°C). Under this conditions shrinkage value of these casings is usually no more than 5-10% in both directions.

Typical dry film based on common commercial aliphatic polyamides (nylons) is too stiff (its Young's modulus or elastic modulus is about 400-500 MPa (40-50 kgf/mm<sup>2</sup>) in both directions and cannot be stretched to desirable level by filling with sausage meat using common stuffing equipment. Therefore before stuffing, in other words, before filling with sausage meat under excess pressure that stretches the casing, it is humidified by soaking in water. After this step a value of its Young's modulus drops to about 200-250 MPa in both directions. Altogether with the appropriate selection of a casing diameter and thickness, this value of the Young's modulus can on one hand provide the possibility of stretching the casing under pressure of sausage meat to desirable level and, on the other hand, to prevent deformation of hung up sausages under their own weight. After the humidifying the casing is stuffed by sausage meat using a stuffing machine (stuffing tube and clipping unit) so that its diameter is increases by 8-12% from its initial value. This stretching is generally enough to provide tough consistence to a sausage. It should be noted that usually casing is stretched unequally in the machine and transverse directions that results in a deformation of a printed image applied onto this casing. Moreover, stuffing and following operations are followed by alternating-sign deformations (decompression-compression) that weakens adhesion between casing surface and printing ink layer and can lead to partial delamination of printed image.

Generally soaking implies contact of water with both external and internal surfaces of a tubular casing. During this operation a contamination of casing internal surfaces directly contacting with food by pathogenic microorganisms or their endospores or by toxic products of their vital activity or decay can take place.

Besides above method of stuffing into tubular casing there is a technique for production of sausage articles by means of FFS type packing machines that transform a tape-form flat film (sheet) by operations of forming from tubular sites, their filling by sausage meat and final sealing of the sausage article. It is obvious that such technique means no stretching of the film, hence sausage cannot be tough unless additional step such as above steam treatment of cooled sausage is performed.

Preliminarily moistened sausage casings that are supplied to sausage-makers are also known. US Pat. № 4 489 295 (published 1990.01.30) disclosed a single-layer oriented tubular casing comprising PA 6 and/or PA 66 and optionally ionomer that was moistened by sterile hot water placed inside of the tube. After saturation with water (that takes some time) the tubular casing was wound on a core and excess of water was removed from it. Then the casing was shirred. Such technique is extremely low-efficient because it greatly elongates production cycle and moreover it requires significant additional working space. In addition it is known that well-moistened polyamide film tends to be stuck together by its adjacent surfaces at their long-time contact for example if it is stored in roll form.

But usually abovementioned moistening operation performed by spraying of cold water onto the tubular casing is combined with shirring step. In this case a tubular casing can contact with water only by its external surface because a time of storage before its stuffing is enough for diffusion and redistribution of moisture in whole volume of the film. The casing should be stored and transposed being packed in a moisture-impermeable container to keep necessary humidity. Meanwhile a long-time keeping of high humidity creates favorable conditions for variant bacteria's or fungi's propagation on an external surface of the casing that can spoil its appearance by their colored colonies.

German patent application № 41 28 081 A1 (published 1993.02.25) disclosed a tubular sausage casing consisting of at least three layers. An external moisture-barrier layer was made of olefinic (co)polymers. A core gas-barrier layer was made of ethylene-vinyl alcohol copolymer (EVOH) or (co)polyamides such as poly (m-xylyleneadipamide) hereinafter referred to as polyamide (or nylon) MXD6 or poly(hexamethyleneisophthalamide/terephthalamide) hereinafter referred to as polyamide (or nylon) 6I/6T. An internal food-contact layer was made of polyamide resin. The casing was moistened in the step of primary tube formation when it was cooled by cold water both from inside and outside and then was wound on a core. In this form moisture soaked an internal polyamide-based layer can be kept for a long time but if the casing was shirred and supplied to sausage-maker in a form of shirred "sticks" it should be packed into additional a moisture-impermeable

package otherwise it would dry up. Consumers should stuff this casing in common manner, i.e. with its stretching.

Russian Patent № 2189146 (published 2002.09.20) disclosed a casing comprising a blended layer made of polyamide and polyamide-block-polyether. Said block-copolymer  
5 has a significant elasticity and can impart these properties to its blends with polyamide. Therefore such casing had reduced Young's modulus and could be stuffed under relatively low pressure of sausage meat and even manually. The casing according to this invention had a level of heat shrinkage value typical for conventional polyamide casings or a little bit more. It was produced according to usual processing scheme for single- or multilayer  
10 polyamide-based casings making. The invention description disclosed neither a typical degree of extension of the invented casing that takes place as a result of its stuffing nor concrete values of sausage meat pressure. It is also impossible to conclude from the description whether invented casing is required to be moistened before stuffing, though all its mechanical characteristics including elastic ones are related to moistened casing.  
15 Meanwhile using of casings with reduced elastic modulus can lead to problems during stuffing and following steps of sausage manufacturing where sausages can be distorted from their usual cylindrical shape to pear-like shape. In addition mentioned block-copolymers are quite expensive and their introducing in casing composition can strongly increase its cost.

20 A third - mixed - approach that implies using of elastic properties and heat-shrinkability simultaneously was disclosed in US Pat. № 5 549 943 (published 1996.08.27). A casing disclose in this Patent showed very high levels of heat shrinkability and shrink force. In this case expanding of sausage meat can be partly (but not completely) suppressed due to high values of compression forces that develops during  
25 sausage cooking. Heat-shrinkable multilayer tubular casing according to this invention had a core layer mainly comprising polyolefin and two peripheral layers mainly comprising polyamide and optionally additional layers from ethylene - vinyl acetate copolymer (EVA) and from EVOH. It was produced according to usual processing scheme for tubular oriented casing production with such thermofixation procedure that implied  
30 heating of dry or wet flattened tubular casing up to 70-90°C. This casing is characterized with shrinkage values of 25-30% in both directions and shrink force values of 96 kgf/cm in the machine direction and of 186 kgf/cm in the transverse direction, measured under its immersing into hot water at 90°C. The casing being dry or moistened was filled with sausage meat by means of stuffing machine. Nevertheless at both cases tough sausages

were prepared. There was no mention in this document about a possibility of filling of this casing without its stretching, though such possibility should have been considered as quite unusual and worthy to be mentioned. Meanwhile above-mentioned drawbacks are intrinsic to this casing and moreover it can cause a problem of clip sliding off from sausage during cooking due to its strong shrinkage. In addition it is impossible to achieve satisfied dimensional stability of a casing during its storage or under operation requiring even moderate heating by above-described manner of thermofixation. For example thermal drying of printing inks applied onto the casing should cause its significant untimely shrinkage.

10 German Patent DE № 28 50 182 (published 1980.06.04) stated that single-layer polyamide casing based on PA 6 or made by orientation drawing and thermofixation at 120°C can be filled with sausage meat with no preliminary moistening and generally without applying pressure (manually). It also stated that in this case it is possible to prepare tough sausages enclosed in non-wrinkled casing only due to its heat shrinkability  
15 (illustrated by no concrete value). However, in description of their later patent (US Pat. № 4 897 295, published 1990.01.30) the same inventors acknowledged that sausage products with satisfactory appearance cannot be made by using the casing according to DE 28 50 182 without its moistening and stretching in its elastic area.

A casing most analogous by its properties and application to the invented casing  
20 was disclosed in US Pat. № 4 303 711 (published 1981.12.01). This biaxially-oriented thermofixed single-layer casing based on aliphatic polyamide, that, after absorption of water, had glass transition temperature from -5 to +3°C, comprised 1-50% of zinc- or calcium-based ionomer and/or EVA and/or modified polyolefin. The last polymers are easy-deformable and non-miscible with polyamide, which have no influence to its glass  
25 transition temperature. Because of these polymeric additives the casing even being dry has high extensibility, comparable with extensibility of moistened polyamide casings. So generally it can be stuffed with sausage meat without preliminary soaking in water. The casing was filled with sausage meat at filling pressures of 0.3-0.6 bar, that are typical for stuffing of moistened polyamide casings, that definitely implies, taking in the account  
30 elasticity of the casing, its stretching under stuffing conditions. During a process of cooking of sausages, polyamide of the casing gained additional elasticity and tough sausages covered by non-wrinkle casing were prepared. The casing according to this invention was made using appropriate thermofixation (120°C for 3-5 minutes) but nevertheless had as inventors stated shrinking properties (also illustrated by no concrete

value). Application of such casing is fraught with risk of undesirable deformation of sausages under their own weight because moisture begins to penetrate into polyamide resin of the casing and reduce mechanical modulus of the casing directly after its stuffing. In addition such casing cannot be used as a film for filling by sausage meat by means of  
5 abovementioned packing machines.

A method of production of polyamide-based tubular casing most analogous to the invented method was disclosed in US Pat. № 4 560 520 (published 1985.12.24). According to the invention a single-layer polyamide film was made by means of a method, comprising the steps of extruding a primary tube of polyamide and simultaneously multi-  
10 axially drawing of the primary tube and subjecting the extruded and multi-axially drawn primary tube to a controlled shrinkage by at least 15% and at most 40% in the transverse and the longitudinal direction in accordance to its transverse and longitudinal dimension after drawing, at temperatures ranging from 90° C to 150°, thereby thermally fixing the primary tube. Thereby made casing had zero shrinkage both at 40 and 80 °C. Therefore it  
15 is impossible to prepare a tough non-wrinkled sausage in such casing unless it has been filled with sausage meat with appropriate stretching.

Thus an object of the present invention is providing of a sausage casing that does not have above-described drawbacks, the casing, which:

- is suitable for being filled with sausage meat by means of automatic stuffing  
20 equipment or packing machines or manually with no preliminary moistening and stretching during its filling and further not requiring additional treatment by hot water or steam after cooling of sausages for preparing tough sausages;
- is characterized by stiffness in wet state high enough to resist deformations appearing in large sausages under their own gravity;
- 25 - does not cause excess stresses during cooking;
- is suitable for being coated with printed image by means of flexography or other high-performance printing technique;
- has desirable mechanical and barrier properties.

Following object of the present invention is providing us with a moderately priced  
30 and high-performance method for production of abovementioned sausage casing.

#### Summary of the Invention

As a result of carried out researches it has been surprisingly found that above-listed objects can be achieved by providing a single- or multilayer synthetic heat-shrinkable sausage casing, comprising at least one polyamide layer made of polyamide

resin (or, in other words, polyamide composition) based on at least one aliphatic (co)polyamide, selected from polyamide 6, polyamide 66, copolyamide 6/66 or their blend, wherein said sausage casing:

- 5       - being ready to be filled, has Young's modulus not less than 300 MPa in the machine direction and not less than 400 MPa in the transverse direction;
- being exposed to air with relative humidity 60% and temperature 25°C for 5 days has shrinkage value that is no more than 2% both in the machine and transverse direction;
- 10      - being exposed to water with temperature 40°C for 2 hours has shrinkage value that is 3-10% in the machine direction and 4-10% in the transverse direction;
- being exposed to water with temperature 80°C for 30 minutes has shrinkage value that is 8-15% in the machine direction and 9-18% in the transverse direction.

In preferable embodiments of the invention said sausage casing has shrinkage value that is:

- 15      - no more than 1% both in the machine and transverse direction on exposure to air with relative humidity 60% and temperature 25°C for 5 days;
- 4-10% in the machine direction and 5-10% in the transverse direction on exposure to water with temperature 40°C for 2 hours;
- 20      - 9-12% in the machine direction and 10-15% in the transverse direction on exposure to water with temperature 80°C for 0.5 hours.

The polyamide layer comprises from 60 to 100% of at least one aliphatic (co)polyamide preferably selected from a group including polyamide 6, polyamide 66, copolyamide 6/66.

25       Besides, the polyamide layer can comprise from 0 to 20% of at least one half-aromatic (co)polyamide selected from the group, including copolyamide 6I/6T, polyamide MXD6, or their blends.

The polyamide layer apart from polyamide composition can comprise from 0 to 20% of at least one resin selected from the group, comprising polyesters, olefin homopolymers, olefin copolymers, ionomers, or their blend.

30       The synthetic casing can be made in a form of tube, half-tube (prepared from flattened tubular film by cutting along one bent line), or sheet (prepared from flattened tubular film by cutting along both bent lines).

The casing according to the present invention can be made by a method, including consecutive steps of single- or multilayer extrudate extrusion, resulting in formation of a

primary tube; orientation drawing of said primary tube, resulting in preparation of an oriented tubular film; thermofixation of said oriented tubular film resulting in preparation of an oriented thermofixed tubular film, wherein said oriented thermofixed tubular film is further subjected to an additional treatment during which said oriented thermofixed tubular film having temperature 60-140°C is drawn by 3-8% in the machine direction and 4-10% in the transverse direction, then cooled, while it is in the drawn (strained) state, to the temperature that is not higher than glass transition temperature of the polyamide composition of the polyamide layer, then is flattened and wound up on a core.

Thermofixation is carried out by heating of the oriented tubular film, being inflated by air in bubble form, up to 130-160°C, preferably up to 135-140°C and its exposure to such temperature lasts for 3-5 seconds. In addition during thermofixation the casing is subjected to shrinkage by no more than 10%, preferably by 5-10% both in the machine and transverse direction.

In a preferable embodiment of the invented method during the said additional treatment the thermofixed oriented tubular casing is drawn by 4-8% in machine direction and by 5-10% in transverse direction.

Besides, the drawing during the said additional treatment is preferably performed at 70-130°C and the casing in the drawn (strained) state is cooled to the temperature not higher than glass transition temperature of polyamide layer matter and preferably not higher than temperature ranging between 45 and 57°C, depending on the composition of the polyamide layer.

The additional treatment of the oriented tubular film can be performed both in the same manufacturing line as steps for production of the oriented tubular film and off-line in a separate apparatus.

The resulted sausage casing can be used *per se*, or be cut along one or both bent lines of flattened tube resulting in making of correspondently one half-tube or two extensive flat films (sheets). In addition the tubular casing can be made in a form of shirred stick.

Manufacturing process of sausage product in the casing according to the present invention consists in filling of the casing with sausage meat virtually under zero pressure (increment of its diameter herein can be no more than 1.5%) resulting in row sausage product, its moisture-heat treatment and following cooling. There is no need in preliminary moistening of the casing and additional heat treatment of the resulted sausage.

The sausage product made in the invented casing according to above making process is tough and non-wrinkled sausage.

### Detailed Description of the Preferred Embodiments

#### 5 **Description of the casing according to the present invention**

In the following description conventional names of polymeric resins and abbreviations are used. Sign “%” means weight percentage based on the total weight of a composition, unless otherwise mentioned or unless another interpretation clearly follows from a context. Hereafter a word, including a prefix put in parenthesis, is use to denote  
10 concept simultaneously meaning concepts denoting by this word with this prefix and without it. For example the word (co)polymer simultaneously means polymer (homopolymer) and copolymer.

Here the terms “ready-to-be-filled casing “ or “sausage casing in the ready-to-be-filled state” mean sausage casing that can be filled with sausage meat directly after its  
15 pulling out of its package with no additional operation. Said package can be impermeable for moisture or another factor depending on concrete structure and form of the invented casing.

The casing according to the present invention in the ready-to-be-filled state has Young’s modulus typical for non-moistened and not-plasticized polyamide-based  
20 casings. In other words it is characterized with significant stiffness, which is absolutely untypical for polyamide-based casings in the instant of their filling. Such casings being preliminarily moistened usually have Young’s modulus that is 1.5-2.5 times lower.

Nevertheless the casing can keep its dimensional characteristic at least being packed into a moisture-impermeable package made, for example, of polyethylene film,  
25 which can provide constancy of air humidity inside of the package at temperature changes during its transportation. Such dimensional stability can be estimate by so called “free shrinkage” measured after exposure of test casing to air to the temperature of 25°C and RH of 60% for 5 days. Under these conditions its free shrinkage does not exceed 2% and preferably 1% both in the machine and transverse direction. Such humidity  
30 conditions can be created in an exsiccator onto bottom of which suitable crystalline salt, for instance, sodium bromide (NaBr) or its saturate aqueous solution is put.

According to the present invention in the casing under high humidity and temperatures about 80-100°C, in other words, under conditions of sausage cooking, a development of shrinking forces takes place, and these forces suppress sausage meat

expanding. If under these conditions shrinkage value is about 8-15% in the machine direction and 9-18% in the transverse direction, a tension stresses of casing on sausage are approximately equal to those, developed by cooking of sausage in conventional polyamide-based casing, which was not additionally treated according to the invented procedure, but was stretched by sausage meat during stuffing in usual manner (i.e. by 10-12%). If the shrinkage values exceed upper values of abovementioned range, it can cause excess tension of the casing connected with risk of its rupture or clip sliding off from sausage. Hereafter this type of shrinkage that is measured after exposure of a casing to water with temperature 80°C for 30 minutes will be denoted as "hot shrinkage". In fact hot shrinkage value of the invented casing is inside above range and is preferably 9-12% in the machine direction and 10-15% in the transverse direction.

Unlike known heat-shrinkable casings, the invented casing has a special feature, namely, significant fraction of the cooking time shrink force developed therein continues to take affect after cooling of sausages. The indispensable condition for activation of this residual shrink force is a significant humidification of the polyamide layer that can be rapidly achieved by sausage cooking (boiling or scalding). Such specific shrink behavior of the invented casing is a result of its additional treatment according to the present invention and gives possibility to prepare taut sausages enclosed therein without the additional heat treatment of the sausages after their cooling. This residual shrink force can be activated at lower temperatures, while other part of shrink force, caused by changes in crystalline phase of polyamide occurring at higher temperatures, is inactive. As it will be shown bellow, activation of the residual shrink force can be achieved by moistening of the invented casing at temperatures from 20 to 50 °C, and inside of above range (20-50 °C) the value of equilibrrious (maximum at a given temperature) shrinkage does not depend on temperature of water, wherein the casing is immerged, but depends on exposure time. At temperatures above 50°C activation of above-mentioned part of shrink force, caused by changes in crystalline phase of polyamide, takes place. In some embodiments of the invented casing said humidification of the polyamide layer at temperatures around room temperature (18-28 °C) takes too much time, therefore the measurement of the shrink properties is reasonable to be carried out at higher temperatures. For example, if the casing has external (or all) layers made of polyamide composition its equilibrrious shrinkage can be measured at 25°C for 1-2 hours. But if it has at least one polyamide layer, enclosed by layers made of moisture-barrier resins, it requires either very long time of soaking of the casing at room temperature or the same

result can be achieved at 40°C for 2 hours, as it will be shown bellow (see Table 3). In the general case it is reasonable to carry out this test at 40°C for 2 hours. This type of shrinkage hereafter will be denoted as «wet shrinkage». In order to prepare taut sausage enclosed into the invented casing and cooked, its shrinkage at these conditions should be 3-10% in the machine direction and 4-10% in the transverse direction. Actually, wet shrinkage values of invented casing are correspondent to these figures, and preferably are 4-10% in the machine direction and 5-10% in the transverse direction.

The invented casing can be both single-layer and multilayer. The casing structure is chosen according to its form, supposed application, storage and transportation conditions and also supposed shelf-life of food product packed therein.

The invented casing in any embodiment comprises at least, one polyamide layer, wherein total percentage of aliphatic (co)polyamides is not less than 60%.

As said polyamides conventional commercially available polyamide resins (nylons) can be used. The nylons are not characterized with such specific features as, for example, high elasticity in non-oriented (cast) state. However such nylons being raw materials for sausage casing certainly should be resistant to prolonged action of hot water, i.e. they should not melt, resolve, disperse or lose their mechanical strength in hot water. Preferable (co)polyamides used for invented casing are nylon 6, nylon 66 or nylon 6/66, which are components of said layer in percentage of 60 to 100%. Semiaromatic (co)polyamides such as copolyamide 6I/6T, polyamide MXD6 in percentage of 0-20% can also be used therein. Said polyamide layer except (co)polyamides can comprise up to 20% of other polymeric resins such as polyesters like, for instance, polyethylene terephthalate (PET), polyethylene terephthalate modified by glycols (PETG), polybutylene terephthalate (PBT), olefin (co)polymers, like, for instance, polyethylenes, polypropylenes, ethylene/propylene (co)polymers (EPR), ionomers etc, or their blends.

The invented casing can include more than one polyamide layer, in this case total thickness of the polyamide layers should be generally at least 40% and preferably at least 45% based on total thickness of the casing. At the same time, total thickness of the casing should be from 18 to 120 µm.

Except described polyamide layer (or layers) the casing can have one or more layers, preferably comprising (co)polyamides other than polyamide components of above polyamide layer, including polyamide 9, polyamide 10, polyamide 610, polyamide 12, polyamide 612, copolyamide 6/66/10, copolyamide 6/66/12, olefin (co)polymers, olefin (co)polymers, grafted by maleic anhydride, polyvinylidene chloride, ethylene/vinyl

alcohol copolymers, thermoplastic polyurethanes. These layers can also comprise blends of above listed resins selected from both same and different polymeric types. Polyamide components of above polyamide layer (layers): polyamide 6, polyamide 66, copolyamide 6/66, copolyamide 6I/6T and polyamide MXD6 can also be the components of these  
5 additional layers in percentage of no more than 40%, for example, as their blend with EVOH. The use of rigid polymeric resins like aromatic polyesters, for example, PET as main raw materials for such separate layers is not desirable in the context of the invention objects, because said resins would impart to the casing stiffness, which cannot be eliminated by humidification.

10 The casing in tubular form can include food-contact layer, made of any thermoplastic resin, approved by its hygienic characteristics for direct contact with food. However, in preferable embodiments, this layer should mainly consist of (co)polyamide or linear thermoplastic polyurethane, because such resins provide adhesion of the casing to meat emulsion that is strong enough. In the most preferable embodiment of the tubular  
15 casing this layer is mostly made of (co)polyamide resin. If material of the layer has no inherent adhesion to sausage meat, this property can be imparted to it by means of known technique, for example, by corona discharge treatment.

If the casing is performed in the form of half-tube or extensive flat film and intended to be filled by means of packing machine, the food-contact layer is made of any  
20 sealable resin approved for direct contact with food, that can be sealed by certain technique at certain machine, providing seam that is strong enough and resistant to the conditions of sausage cooking. It is preferable that said sealable resin should have melting temperature in the range of 101-160°C. Suitable resins comprise olefin (co)polymers, ionomers, and also thermoplastic polyurethanes or polyamides, for  
25 example copolyamide 6/66/12, sold by EMS Co (Switzerland) under trademarks Grilon CF6N or Grilon CF7N. Preferably this layer is made of ionomer, EVA, metallocene polyethylene (linear low density polyethylene, hereafter LLDPE, linear very low density polyethylene, hereafter LVLDPE, etc), ethylene/(meth)acrylic acid copolymer or PVDC or their blends. In order to impart suitable meat-adhesion to said layer one can use  
30 techniques known from prior art, namely plasma treatment, corona treatment or irradiation by UV-rays, X-rays,  $\gamma$ -rays, e-beams, etc. The preferable method of such treatment is the corona treatment technique. If the film or half-tube is intended to be sealed with an overlap during formation of the package, an air-contacting layer should be also made of sealable resin.

For the casing disclosed in the present invention prolonged contact of the polyamide layer (layers) with humid environment air is undesirable because it affects the dimensional stability of the casing. Therefore, if such casing is intended to be stored and transposed without moisture-impermeable container, the polyamide layer (layers) is preferably insulated from environment air by layers made of moisture barrier resins. If the casing is supplied to a consumer as a tubular film wound in roll form, only one external (air-contacting) moisture-impermeable layer is enough. If the tubular casing is supplied to a consumer as a shirred stick and air has free access inside the tube, at least the thickest polyamide layer should preferably be surrounded by two moisture-impermeable layers. In order to decrease sensitivity to humidity of the polyamide layer(s) semiaromatic (co)polyamides such as copolyamide 6I/6T and/or polyamide MXD6, increasing glass transition temperature, and also polyolefins, ionomers, or polyesters like PET, PETG and PBT can be added therein.

As raw materials for moisture-impermeable layer(s) polypropylene, ethylene/propylene copolymer comprising at least 80 mol. % of propylene units, low density polyethylene (LDPE), high density polyethylene (HDPE), metallocene LLDPE, metallocene LULDPE, EVA, and also products of maleic anhydride grafting onto said resins, PVDC and blends of all above polymers are preferably used.

Any layer or a few layers of the casing according to the present invention can additionally comprise from 0 to 10% of at least one low molecular weight additive, selected from a group, comprising colorants, pigments, fillers, lubricants, processing aids, foaming additives, slipping additives, matting additives.

The casing according to the present invention can be used for packing of sausage products with various shelf-life. For example a casing for short-time storage can be single-layer. A casing for more prolonged storage should prevent drying-induced loss of weight of the food content and protect it from atmospheric oxygen penetration, causing its spoilage (fat rancidity, etc). Therefore such casing comprises layers made of resins having oxygen-barrier properties.

Polyamide resins such as polyamide 6 or polyamide 66 have enough high oxygen-barrier properties; however their oxygen permeability strongly depends on their humidity. Therefore it is preferable that during storage of a sausage product, at least thickest (main) of polyamide layers is relatively dry. It can be achieved if the main of polyamide layer is in contact with environing air and insulated from humidity of sausage meat by layer of moisture-impermeable resin. However, in this case, the condition of

polyamide layer insulation from environing air, quite desirable for casing storage, is not achieved. Therefore it is preferable that the main polyamide layer at the same time is insulated both from environmental air and humid sausage meat by layers made of moisture-impermeable resin, but in such a way that water vapor transition rate (WVTR) of the first of said layers (between polyamide and air) is greater than of a second one (between polyamide and meat). If the said moisture-impermeable layers are made of resins having similar values of moisture permeability coefficient, the last condition can be achieved by proper choice of their thickness, i.e. total thickness of moisture-impermeable layers placed between polyamide and air is far less than the thickness of moisture-impermeable layers placed between polyamide and meat. Besides that, one can reduce the sensibility to humidity of oxygen-barrier properties of the polyamide layer, mainly made of aliphatic polyamide, such as polyamide 6 or polyamide 66, by adding therein up to 20% of semiaromatic (co)polyamide 6I/6T or polyamide MXD6. Except the polyamide layers the oxygen penetration protection can be provided by additional layers from such oxygen-barrier resins as PVDC or EVOH. Oxygen permeability of PVDC layer does not depend on its humidity; therefore a position of this layer in casing structure does not matter. If it is desirable that both polyamide-based layer and PVDC layer simultaneously act as oxygen barrier, moisture impermeable PVDC layer should be placed closer to sausage meat, than the polyamide layer. EVOH resins are moisture-sensitive too, and the dependence on humidity of their oxygen-barrier properties is even stronger than that of polyamide resins. Therefore the EVOH layer in the filled sausage casing is preferably placed as close to air as possible, but it should not be external.

For bonding of layers, made of resins having different chemical nature and not having inherent mutual adhesion, adhesive layers from olefin (co)polymers grafted by maleic anhydride can be used. The best interlayer adhesion is achieved if the type of olefin (co)polymer for moisture-barrier layer and type of backbone of grafted resin for layer providing adhesion between said barrier layer and other layer (polyamide, EVOH, etc) are the same. Such adhesive resins can be either the only component of the separate adhesive layer or one of components of mixed layer, also comprising relative to adhesive resins olefinic polymer and having both barrier and adhesive function.

The casing can be performed in the form of tube, half-tube, or extensive flat film (sheet). It depends on whether it is intended to be stuffed by means of stuffing equipment (tube) or to be filled in a packing machine (half-tube or sheet). Upon the casing in any of said form a printed image can be applied, and a tubular casing can also be shirred.

Besides, the printed image can be applied onto invented casing by means of flexography or other high-performance printing technique.

Possible variants of structure of the casing according to the present invention are shown in the Tables 1 and 2.

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Table 1. Some possible embodiments of the casing according to the present invention, used in the tube form to be stuffed by means of stuffing equipment.

Casing structure (from meat to air - LTR)		Need of packing for storage	Need of corona treatment	Barrier properties in respect of	
				O <sub>2</sub>	H <sub>2</sub> O vapor
PA		yes	no	good	moderate
PA PO+A (PA A PO)	roll	no	no	poor	good
	shirred stick	yes	no		
PO A PA		yes	yes	very good	good
PO1 A PA PO2+A (PO1 A PA A PO2) <sup>a b</sup>		no	yes	good	good
PA PO1+A PA PO2+A (PA PO1 A PA PO2+A) (PA PO1+A PA A PO2) (PA PO1 A PA A PO2) <sup>a</sup>	roll	no	no	good	good
	shirred stick	yes			
PA PO1+A PA EVOH PO2+A (PA PO1 A PA EVOH PO2+A) (PA PO1+A PA EVOH A PO2) (PA PO1 A PA EVOH A PO2) <sup>a</sup>	roll	no	no	very good	good
	shirred stick	yes			
PA A PVDC A PA PO+A (PA A PVDC A PA A PO)	roll	no	no	very good	very good
	shirred stick	yes			

PA – polyamide composition, comprising mainly polyamide 6, polyamide 66 or  
10 copolyamide 6/66 or their blend and, optionally, (co)polyamide 6I/6T and/or MXD6;

PO – polyolefin (PP, CPE, LDPE, HDPE, LLDPE, LVLDPPE), EVA, ethylene/(meth)acrylic acid copolymer, ionomer or their blend;

A – adhesive resin, preferably, polyolefin, grafted by maleic anhydride;

PO+A – blend of PO and A;

5 EVOH – ethylene/vinyl alcohol copolymers with 32-44 mol % of ethylene units;

PVDC – thermoplastic copolymer of vinylidene chloride with vinyl chloride and/or vinyl acetate with 75 -90 mol % of vinylidene chloride units;

In symbols PO1, PO2 – a figure corresponds to a sequence number in the direction from meat to air of polyolefin layer in a polymer film, if such layers are more than one;

10 <sup>a</sup> Layer PO1 (PO1+A) is preferably far thicker than a layer PO2 (PO2+A).

<sup>b</sup> Variant of food contact layer of PO1+A type is out of consideration here because adhesive resins often have no approval for direct contact with food.

15 Table 2. Some possible embodiments of the casing according to the present invention, used in the half-tube or extensive flat film (sheet) form to be filled by means of packing machine.

Casing structure (from meat to air - LTR)	Relative position of sealed edges	Sealing method	Barrier properties in respect of	
			O <sub>2</sub>	H <sub>2</sub> O vapor
PO  A  PA <sup>a b</sup>	butted	pressure contact	very good	good
PVDC A PA	butted	pressure contact or induction	very good	very good
PO1 A PA PO2+A (PO1 A PA A PO2) <sup>a b</sup>	butted or overlapped	pressure contact	good	good
PO1 A PA EVOH PO2+A <sup>a b</sup>	butted or overlapped	pressure contact	very good	good
PVDC A PA PO+A (PVDC A PA A PO) <sup>c</sup>	butted	pressure contact or induction	very good	very good
	overlapped	pressure contact		
PVDC1 A PA A PVDC2	butted or overlapped	pressure contact or induction	very good	very good

All the casings require the special treatment for providing adhesive properties in respect of food contents and all of them except the first two do not need additional packaging.

PA – polyamide composition, comprising mainly polyamide 6, polyamide 66 or copolyamide 6/66 or their blend and, optionally, (co)polyamide 6I/6T and/or MXD6;

5 PO – polyolefin (PP, CPE, LDPE, HDPE, LLDPE, LVLDPPE), EVA, ethylene/(meth)acrylic acid copolymer, ionomer or their blend;

A – adhesive resin, preferably, polyolefin, grafted by maleic anhydride;

PO+A – blend of PO and A;

EVOH – ethylene/vinyl alcohol copolymers with 32-44 mol % of ethylene units;

10 PVDC – thermoplastic copolymer of vinylidene chloride with vinyl chloride and/or vinyl acetate with 75 -90 mol % of vinylidene chloride units;

In symbols PO1, PO2 (PVDC1, PVDC2) – a figure corresponds to a sequence number in the direction from meat to air of polyolefin (PVDC) layer in a polymer film, if such layers are more than one;

15 <sup>a</sup> Layer PO1 (PVDC1) is preferably thicker than a layer PO2 (PO2+A, PVDC2).

<sup>b</sup> Variant of food contact layer of PO1+A type is left out of consideration because adhesive resins often have no approval for direct contact with food.

<sup>c</sup> The thickness of PVDC layer is selected by such way that it is characterized with WVTR far lower than WVTR of PO (PO+A) layer.

20

Description of the method of production of the casing according to the present invention

The first three steps of the method for production of the casing according to the present invention including extrusion of single- or multilayer extrudate as a primary tube, the orientation drawing of the primary tube resulting in production of an oriented tubular film and thermofixation of the oriented tubular film tube resulting in a production of a thermofixed oriented tubular film disclosed above are typical for production method of conventional sausage casings, and the method is usually limited by aforesaid.

30 Conventionally used following additional operations for applying a printed image and/or shirring can only change appearance or shape of the casings, but not their properties.

A cast “workpiece” of the tubular casing is formed by the extrusion. In this stage extrudate, formed by means of a circular die hole is cooled as fast as possible (quenched) to be transformed into so called primary tube. Water is preferably used as cooling

medium. Such technique prevents nylon crystallization and facilitates following orientation drawing.

Heating of the primary tube to about 70-80°C precedes the drawing stage. This step can be performed in a convection tunnel oven, infrared tunnel oven or a bath with hot water. The biaxial drawing of tubular casing is performed by means of inflating of primary tube by air, which remains entrapped therein by two pairs of driving nip rollers, to transform it into a bubble (secondary tube or secondary bubble) and its simultaneous drawing in the machine direction by means of said two pairs of nip rollers, due to a difference in rotation speeds of the rollers in each pair: rollers of second (in the direction of the film motion or downstream) pair rotate faster than of first one. This method is also called "entrapped bubble technique". At this operation the primary tube is drawn 2-3 times (according to ratio of the rollers rotation speeds) and its diameter of is usually increased 3.5-4 times. The draw operation results in a tubular film, having the polyamide layer in unstable oriented state, characterized with presence of a plurality of small and imperfect crystallites tending to recrystallization.

Thermofixation of the oriented tubular film is preferably performed by exposure of this film as an air-inflated bubble (tertiary tube or tertiary bubble) to temperatures of 130-160°C, and preferably of 135-140°C for 3-5 seconds. For the heating of the bubble a convection oven or infrared heater are preferably used. Thermofixation by means of treatment of hot water or steam known from prior art (see for example European Patent No EP0974452) is not acceptable, because in the context of the invention objects the polyamide layer should be dry after thermofixation. During thermofixation the oriented tubular film is shrunked by up to 10% and preferably by 5 to 10%, both in the transverse direction and in the machine direction due to selected excess pressure in the bubble and speed ratio of pairs of nip rollers at the beginning and at the end of the bubble. By adjusting the temperature and time of thermofixation, and also degree of "bubble shrinking", one can prepare tubular films with various values of "hot" shrinkage, which can be measured if production of the invented casing is performed by means of two separate apparatus. The fraction of the "hot" shrinkage is also inherited to a ready-to-use casing according to the present invention after the additional treatment and it is approximately equal to difference between its "hot" shrinkage and its "wet" shrinkage. However if thermofixation temperature is equal to or lower than 120°C, the resulting casing is characterized by inferior dimensional stability and high values of heat shrinkage. The proper thermofixation leads to a formation of stable crystalline phase of polyamide.

Its stability is manifested by absence of tendency to recrystallize either spontaneously under storage conditions or under the action of moderate tensile deformations for instance of those, that take place at the casing filling. After thermofixation step according to conventional techniques the flattened tubular casing is cooled to a room temperature or a little bit higher and wound on a core.

The casing prepared by means of said first three stages, i.e. according to conventional production process, is generally characterized by virtually zero “free” shrinkage during the storage at a room temperature, “wet” shrinkage value of about 0 to 1.5 in both directions and “hot” shrinkage value of about 5 to 8% in both directions.

The following stage is the essence of the invented method for production of the casing according to the present invention. After thermofixation stage the tubular casing with temperature, exceeding glass transition temperature of polyamide composition of the above polyamide layer, is drawn by 4-10% in the transverse direction and by 3-8% in the machine direction, preferably by 5-10% in the transverse direction and by 4-8% in the machine direction, with following cooling in the drawn (strained) state to temperature below glass transition temperature of polyamide composition. The range of glass transition temperatures of polyamides 6 and 66 (dry), mainly composing polyamide composition, is the temperature area between approximately 47 and 60°C (Nylon Plastics Handbook ed. by Melvin I. Cohan, Cincinnati: Hanser/Gardner Publication, Inc, 1995, сtp. 147). The glass transition point (temperature) is usually understood as the lowest value of this range. Addition of semiaromatic (co)polyamides 6I/6T and/or MXD6 into polyamide composition can result in an increasing of glass transition temperature of polyamide composition. For example, adding of 20% copolyamide 6I/6T (trademark Selar® PA 3426, from DuPont) into polyamide 6 increases glass transition temperature of the latter by 10°C (see, for example, a brochure «DuPont™ Selar® PA3426 Blends With Nylon 6» - URL: [http://www2.dupont.com/Selar/en\\_US/assets/downloads/selar\\_pa3426\\_nylon\\_blends.pdf](http://www2.dupont.com/Selar/en_US/assets/downloads/selar_pa3426_nylon_blends.pdf). Date: 2009.03.24).

At the additional stage of the invented method a value of drawing temperature (above the glass transition temperature of polyamide composition) generally does not matter and can be selected mainly according to economic considerations. The drawing temperature is in the range 60-140°C, but preferably 70 to 130°C. If polyamide composition comprises no semiaromatic (co)polyamides such as 6I/6T and/or MXD6, the drawn tube is preferably cooled to temperature not higher than 47°C, preferably to

temperature 45°C, but if said composition comprises 20% of polyamide 6I/6T, the tube can be cooled to temperature not higher than 57°C, preferably to temperature 55°C. The final cooling of the casing to room temperature can be performed after flattening of the tube, for example, by means of a chilling roller. In a preferable embodiment the biaxial draw of the casing is performed by means of its simultaneous inflating by air in bubble and drawing in the machine direction by means of two pairs of driving nip rollers by the same manner that has been disclosed above for stage of the orientational draw. This step of the production process can be performed both in the same manufacturing line as steps for production of the oriented tubular film and in a separate apparatus. This apparatus comprises sequentially arranged unwinding roller, a heater, two pairs of driving nip rollers, a cooling device and a winding roller. Such apparatus can compose a manufacturing line together with shirring machine, operating at constant rate of casing feed. A tunnel convection oven or a tunnel infrared oven can be used as the heater. The heater can be set between the unwinding roller and the first pair of the nip rollers to heat a flattened tube, or first and second pair of the nip rollers to heat the bubble. The cooling of the inflated bubble is preferably performed with stream of cold air by means of a ring-shaped air-blower. If the step of the additional drawing is performed in-line with forgoing steps for production of conventional tubular casing, there is no need in additional heater, because the casing is still hot enough after thermofixation step, but other units should be set. The additional stage according to the present invention takes short time, because the additional bubble has a length not exceeding 3 meters, even if it is built-in into high-performance production line.

The performance of the additional drawing out of main production line is preferable if, for example, the tubular casing has an external (air-contact) polyamide layer and a printed image is intended to be applied upon it by using printing inks dissolved in lower alcohols or water. If the casing has been additionally drawn, such procedure can cause its local shrinkage and distorting of its regular cylindrical shape. In this case the application of the printed image preferably foregoes the additional drawing step and a design of this image should be developed with a glance to the preprogrammed draw-induced distortions.

If an ultimate consumer needs the tubular form of the casing it can be supplied either as rolls or as shirred sticks. In the last case it can be produced from a wound flattened tube (roll) into shirred sticks by means of well-known technique of shirring.

If an ultimate consumer needs a film intended to be formed, filled with sausage meat and sealed by means of SSF packing machines of horizontal or vertical type, the initial tubular casing according to the present invention can be performed into a half-tube or an extensive flat film by cutting along one or both bent lines of flattened tube correspondently. In the last case two rolls of the flat film are produced. Such films with applied printed image can be made by both applying the image onto these films *per se* and by applying the image onto the invented tubular casing and its following cutting.

Besides, the extensive flat film can be produced out of the tubular casing, having twice smaller thickness, by bonding of two halves of flattened tubular casing by suitable glue comprising no solvent and require no heat to be applied, for instance, UV-curable glue. Such operation can be performed by means of an apparatus comprising sequentially arranged unwinding roller, two pairs of driving nip rollers, UV-lamp and a winding roller, wherein UV-curable glue is loaded into a bubble between the pairs of driving nip rollers and subjected to curing in a thin layer between the halves of flattened tubular casing.

15

**Filling of the casing by means of stuffing equipment.** As it was mentioned above, the conventional process for stuffing with sausage meat of plastic polyamide-based casings comprises a stage of their preliminary soaking with water that usually takes 0.5 to 1 hour. There is no need in this procedure for the casings according to the present invention. Then said stuffing of the casing by means of stuffing machine composed by stuffing tube and clipping unit is performed. It is usually accompanied with stretching of the tubular casing diameter by 10-12%. Filling of the invented casing results in no or very little (by 0.5 to 1.5%) extension of its diameter. Actually, according to a wish of a sausage-maker the casing can be stretched at stuffing so far as it is allowed by stuffing equipment without damage to the end product quality and without excess deterioration of the equipment. Nevertheless the invented casing is preferably filled with no stretching. Right after stuffing the sausages can be slightly flabby by touch, except the case when the main polyamide layer directly contacts with sausage meat and the contacts with humid medium for very short time causes shrinkage of the casing, its tension and compression of its content. The final cooked sausage product enclosed into the invented casing in any embodiment is a tough stuffed log (stick).

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**Filling of the casing by means of packing machine.** In the case of the filling of the casing by means of packing machine, the use of casing with maximum “wet” shrinkage is preferable. Filling is performed according to usual processing scheme.

5           **Cooking of the sausage product.** In both cases of filling of the casing the cooking of the sausage product is performed according to usual processing scheme. Scalding, boiling and even retorting (autoclaving ) processes can be used. During these water and heat treatments water penetrates into polyamide layer and, from this moment the forces tensing the casing around the sausage meat begin to act.

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**Cooling of the sausage product.** Cooling of the sausage product should be performed in such a way, that the polyamide layer remains humid for all cooling time period. Generally, cold water sprinkling or showering are the preferable methods of cooling. If the main polyamide layer of multilayer casing according to the present  
15 invention is a core layer, one may use cooling by cold air stream with caution, but if this layer is food-contacting, the cooling by cold air can be used with no doubt.

**Following operations.** There is no need in following operations. Sausage products can be put into a refrigerator right after said cooling.

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**Sausage product enclosed in the casing according to the present invention.** Such sausage product is a tough log without wrinkles. Its following use depends on barrier properties of the casing which are determined by its composition and structure. For example, sausage product enclosed in the casing, comprising food-contacting polyamide  
25 layer without additional oxygen barrier layers is preferable to use for following fabrication of either sausage slices enclosed into a package made of a barrier film, preferably into barrier vacuum bag. Other types of the product can be stored in a refrigerator for a time period approved for a certain type of the casing by Medical Authorities basing on hygienic regulations.

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          Following examples demonstrate some possible embodiments of the casing according to the present invention, and also the method of production thereof. These examples have only illustrative character and they unconditionally do not cover the total scope and spirit of the present invention, disclosed by its claims.

**Examples of the casing and its characteristics.****Comparative example 1.**

The casing according to this example was a tubular 5-layer casing having diameter  
5 of about 65 mm, wall thickness of about 42-45  $\mu\text{m}$  and sequence of layers (from meat to  
air - LTR):

PA1 | A | PE1 | A | PA2,

with relative thicknesses of the layers: 15% | 3% | 25% | 3% | 54%,

wherein PA1 was a blend of 60 % PA 6 (trademark Ultramid B4), 35% PA 66 (trademark  
10 Ultramid A4) (both polyamides from BASF GmbH, Germany) and 5% PA 6I/6T  
(trademark Selar® PA 3426 from DuPont Co, USA);

A was adhesive polymer Modic 603 from Mitsubishi Chemical Co (Japan);

PE1 was LDPE (trademark 15803-020 from Kazanorgsintez, Russia);

PA2 was blend of 55 % PA 6 (trademark Ultramid B4), 40% PA 66 (trademark Ultramid  
15 A4) and 5% ionomer (trademark Surlin 1652 from DuPont Co, USA).

This casing was manufactured according to first three steps of above method for  
production thereof. Granulated resins were loaded into hoppers of five extruders of  
multilayer extrusion apparatus, according to above sequence of layers. Said resins were  
20 melted in these extruders and their melts were transferred into extruding head where  
melted sequence of 5 layers was formed. Then this 5-layered extrudate was extruded  
through a circular die hole as a melted tubular workpiece into a bath with cold water. The  
workpiece was chilled and solidified transforming into a multilayer primary tube, having  
diameter of about 20 mm and thickness of about 320  $\mu\text{m}$ . Then the primary tube was  
25 transferred by a set of pairs of driving nip rollers into the tubular IR-heater, where it was  
heated to a temperature of 80°C, and then it was biaxially oriented by a factor of 2.5 in the  
machine direction and 3.7 in the transverse direction by the described above  
inflating/drawing technique. Following thermofixation of the resulted oriented film was  
performed by heating of air-inflated tertiary bubble at temperature of 135-140°C for 3-5  
30 sec by means of tubular IR-heater (of tunnel type). During thermofixation the diameter of  
the tertiary bubble was maintained constant of 66-67 mm, but it was allowed to shrink in  
the machine direction by a factor of 1.10 due to corresponding adjustment of rotation  
speeds of the rollers in said two pairs limiting bubble. After thermofixation the resulted  
tubular casing was flattened, cooled to temperature of 25°C and wound on a core as a roll.

Characteristics of the casing are shown in Table 3. After the casing sampling, the roll was packed into polyethylene bag for following transportation to a shirring section.

**Example 1.**

The casing according to this example was made by treatment of the casing, produced according to comparative example 1, at a separate apparatus. A printed image by inks containing ethanol as solvent was applied upon the casing, produced according to comparative example 1. Then a roll of this casing was set in the unwinding roller of an apparatus with total length of 1.5 m comprising except said roller sequentially arranged an IR-heater equipped with flat IR-radiators, first pair of driving nip rollers, a cooling ring-shaped air-blower, second pair of driving nip rollers, chilling roller and a winding roller. The casing was passed through the space between IR-radiators, both pairs of the rollers (unclenched) and the ring of the air-blower; then the first pair was clenched; the tubular casing was inflated to diameter of 72-73 mm; the second pair was clenched; the flattened tubular casing was passed through the chilling roller and its end was bonded on a core set in the winding roller. After that, compressed air was supplied to the blower and rotation of all driving rollers was switched. Rotation speed ratio between the rollers of second and first pair was 1.04; rotation speeds of the unwinding roller and rollers of the first pair on the one hand, and winding roller and rollers of the second pair on the other hand were equal.

During the running of the casing through the apparatus the temperature of the inflated bubble in an area between the first pair and the beginning of a cooling zone was 70 to 80°C and 45°C after this zone. After passing through the chilling roller, the flattened tube had temperature of 25°C. The last piece of the casing with a length of 1.5 m was cut and thrown away. The treated casing has diameter of 71.5 mm (flattened tube width was 112-112.5 mm) and thickness of about 35 µm. Characteristics of the casing are shown in Table 3. After sampling of the casing, the roll was packed into polyethylene bag for following transportation to a shirring section.

**Comparative example 2.**

The casing according to this example was a tubular 7-layer casing having diameter of about 65 mm, wall thickness of about 42-45 µm and sequence of layers (from meat to air - LTR):

PA1 | A | PE2 | A | PA1 | A | PE3

with relative thicknesses of the layers: 13% | 3% | 29% | 3% | 39% | 3% | 10%,

wherein compositions of the layers PA1 and A were identical to those of comparative example 1,

PE2 was blend of из 80% PE1 (according to example 1) and 20% of brown pigment concentrate with percentage of the pigment of about 48% (trademark 1340 Brown from  
5 A. Schulman Inc., Germany),

PE3 was LVLDPPE (trademark AFFINITY PL 1845G from Dow Chemical Co.).

The casing was fabricated in the same manner as in Comparative Example 1, except that 7-layered head was used for forming of the multilayer extrudate and during thermofixation at 135-140°C the casing was allowed to shrink not by 10% only  
10 lengthwise, but by 5% in relation to dimensional parameters of the secondary tube, both in the transverse direction, and in the machine direction. Resulted casing had thickness of about 40.5 μm and diameter of 68-68.5 mm (flattened tube width was 107-107.5 mm). After sampling of the casing, one roll was packed into polyethylene bag and then was transported to a printing section in order to apply an image by UV-curable printing inks  
15 onto the casing. One more roll was packed into polyethylene bag and then was transported to the warehouse. Characteristics of the casing are shown in Table 3.

### **Example 2.**

The casing according to this example was made in the same manufacturing line as that which was used for producing of casings made by comparative examples 1 and 2, but  
20 characterized with additional assembly of units placed in the end of the line directly before chilling roller, wherein said assembly comprised sequentially arranged a zone for free running of the flattened tubular film, first pair of driving nip rollers, two cooling ring-shaped air-blowers and second pair of driving nip rollers. Such reconstruction resulted in increasing of the line length by 3 meters. The start of the producing process for this casing  
25 was identical to that of comparative example 1, but the finish of thermofixation stage and leaving the second of pairs of driving nip rollers, limiting the tertiary bubble, the flattened tubular film was passed through the free running zone and both pairs of driving nip rollers; and this pair was clenched. Then the tubular film was inflated to diameter of about 73 mm, said second pair was clenched and after that the flattened tubular casing was passed  
30 through the chilling roller and its end was bonded on a core set in the winding roller. Compressed air was supplied to the blowers and the casing began to wind on a core. Rotation speed ratio between the rollers of second and first additional pair was 1.05; temperature of the film before entering into the first pair was 105 °C, 60°C after the first blower, 48°C after the second one and 27 °C after passing through the chilling roller.

Resulted casing had a thickness of about 35  $\mu\text{m}$  and diameter 71.5 mm (flattened tube width was 112-112.5 mm). After sampling of the casing, one roll was packed into polyethylene bag and then was transported to a printing section in order to apply an image by UV-curable printing inks onto the casing. One more roll was packed into polyethylene bag and then was transported to the warehouse. Characteristics of the casing are shown in Table 3.

#### **Comparative example 3.**

The casing has sequence of layers identical to that of Example 2. The casing was fabricated in the same manner as in Comparative Example 1, except that thermofixation was performed at 120°C for 2 seconds. The resulted casing had the same diameter and the same thickness, that the casing by comparative example 1.

#### **Comparative example 4.**

The casing has sequence of layers identical to that of Example 1. It was fabricated in the same manner as in Comparative Example 1, except that to thermofixation at 90°C for 2 seconds was subjected a flattened tube. In the machine direction the casing was allowed to shrink by 10%. The resulted casing had a width of flattened tube of 98-99 mm and wall thickness of about 47  $\mu\text{m}$ . After sampling of the casing, one roll was packed into polyethylene bag and then was transported to a printing section in order to apply an image by UV-curable printing inks onto the casing. Characteristics of the casing are shown in Table 3.

#### **Test of printed image stability**

In 5 hours after fabrication of the casings according example 2 and comparative examples 3 and 4, printed images based on UV-curable inks were applied onto their surfaces by means of machine for flexographic printing. After that rolls of the casings were packed in polyethylene film bags and aged for 2 weeks at temperature of 25°C. Then the bags were unsealed and opened and conditions of the rolls and printed images were evaluated. The casings according to the present invention withstood the test: the tension of casing in the rolls was similar to initial one (directly after the application of the printed images). An ink layer of printed image was undamaged.

The roll of the casing according to comparative example 3 was manifestly compressed due to tension of the casing, but ink layer was also not damaged.

The roll of the casing according to comparative example 4 was strongly compressed especially in the central part of its cylindrical surface. Inspection after

unwinding of the roll showed that the ink layer was partly transferred onto opposite half of the flattened tube contacted with it.

Table 3. Basic characteristics of the casings

Example, №	Young's modulus, MPa		Shrinkage, %											
	long.	trans.	In air at 25°C and RH 60%, 2 weeks		In water at 25°C, 2 hours		In water at 25°C, 3 days		In water at 40°C, 2 hours		In water at 60°C, 0,5 hours		In water at 80°C, 0,5 hours	
0			long.	trans.	long.	trans.	long.	trans.	long.	trans.	long.	trans.	long.	trans.
1 comp.(dry)	513	514	<1	<1	1	1	1	1	1	1	3	4	4	5
1 comp.(wet)	196	261	N.	N.	N.	N.	N.	N.	N.	N.	N.	N.	N.	N.
1 (dry)	521	532	1	1	7	8	8	9	8	9	11	13	12	14
2 comp.(dry)	382	345	<1	<1	1	1	1	1	1	1	5	6	7	8
2 comp.(wet)	215	247	N.	N.	N.	N.	N.	N.	N.	N.	N.	N.	N.	N.
2 (dry)	339	567	<1	<1	1	1	5	4	4	5.5	9	10	10	12

Young's moduli were measured by means of test apparatus AUTOGRAPH AGS from Shimadzu Co;

Aging of the samples on air at 25°C and RH 60% for measurement of their free shrinkage was performed in an exsiccator with sodium bromide (NaBr) put on its bottom, wherein the exsiccator was placed in a thermostat maintaining above temperature.

Tests of shrinkage in water at various temperatures (except the variant at 25°C for 2 hours) were then prolonged to double aging period for every temperature, but showed no significant change of dimensional parameters of the casing .

RH – relative humidity, long. – longitudinal; trans. – transversal; comp. – comparative;

(dry) – before the measurement of Young's modulus, the samples were not soaked in water

(wet) – before the measurement of Young's modulus, the samples were not soaked in water at 25°C for one hour;

N. – измерение не проводилось.

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### **Examples of application of the casings.**

#### **Filling at stuffing manufacturing line**

The casings according to examples 1 and 2, and also to comparative examples 1, 2 and 3, bearing printed image and shirred were transported to a sausage-making factory wherein they were filled with sausage meat in the manner that diameters of the resulted sausages exceeded the nominal ones by no more than 0.5%. The filling was performed by means of stuffing line, comprising the stuffer HANDTMANN VF618 from Albert Handtmann Maschinenfabrik GmbH & Co. KG and automatic clipping device FCA-3463 from Poly-Clip System GmbH & Co. KG.

Then the sausages were transferred into a cooking chamber, wherein they were subjected to steam treatment (steam temperature was about 100°C) until the temperature of the central area of a sausage achieved 70°C that took about 40 minutes. During the cooking casings of all sausages looked tight. After that all the sausages were cooled by cold water sprinkling (showering) and a part of the sausages enclosed in casings according to examples 1 and 2 was cooled by cold air stream. Resulted sausages cooled by water were taut if only they were enclosed in casings according to the present invention. In the case of cooling by air, only sausages enclosed in casings according to present example 2 were taut. In all other cases sausages were flabby and wrinkled.

**Imitation of filling by means of packing machine**

Outer surfaces of the tubular casings according to example 2 and comparative examples 2 and 3 were treated by corona discharge until surface energy of 80 dyne/cm was achieved. Then pieces of the casings with length of about 40 cm each were cut along one of bent lines of flattened tubes and resulted half-tubes and were rearranged in such manner that its surfaces comprising polyolefin layer were faced to each other and butt-sealed by means of contact welding with seam of 2 mm width. After that a casing film on one butt end of each resulted turned inside out tube was pleated and fixed by a clip from thick aluminum wire. Then the tube was manually filled with sausage meat and fixed by a clip from second butt end. Resulted sausages were cooked in boiling water for 30 minutes and then were cooled in cold flowing water. As a result sausage enclosed in casings according to example 2 proved to be tout an unwrinkled but the casings according to comparative examples 2 and 3 proved to be wrinkled on the surface of sausages. Moreover, after cutting of the sausage, under the casing according to comparative example 2, meat gel (so called "cook out") was found. Hence, above tests showed that only casings according to the present invention met the set objects.

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## CLAIMS

1. A single- or multilayer synthetic heat-shrinkable sausage casing, comprising at least one polyamide layer made of polyamide resin based of at least one aliphatic (co)polyamide, selected from polyamide 6, polyamide 66, copolyamide 6/66 or their  
5 blend, wherein said sausage casing:
- i. being ready to be filled, has Young's modulus not less than 300 MPa in the machine direction and not less than 400 MPa in the transverse direction;
  - ii. being exposed to air with relative humidity 60% and temperature 25°C for 5 days has shrinkage value that is no more than 2% both in the machine and  
10 transverse direction;
  - iii. being exposed to water with temperature 40°C for 2 hours has shrinkage value that is 3-10% in the machine direction and 4-10% in the transverse direction;
  - iv. being exposed to water with temperature 80°C for 30 minutes has shrinkage value that is 8-15% in the machine direction and 9-18% in the transverse  
15 direction.
2. The synthetic casing according to claim 1, wherein said synthetic casing being exposed to air with relative humidity 60% and temperature 25°C for 5 days has shrinkage value that is no more than 1% both in the machine and transverse direction.
3. The synthetic casing according to claim 1, wherein said synthetic casing being  
20 exposed to water with temperature 40°C for 2 hours has shrinkage value that is 4-10% in the machine direction and 5-10% in the transverse direction.
4. The synthetic casing according to claim 1, wherein said synthetic casing being exposed to water with temperature 80°C for 30 minutes has shrinkage value that is 9-12% in the machine direction and 10-15% in the transverse direction.
- 25 5. The synthetic casing according to claim 1, wherein said polyamide layer comprises from 60 to 100% of at least one aliphatic (co)polyamide.
6. The synthetic casing according to claim 5, wherein said polyamide layer comprises from 0 to 20% of at least one half- aromatic (co)polyamide selected from the group, comprising copolyamide 6I/6T, polyamide MXD6, or their blend.
- 30 7. The synthetic casing according to claim 5, wherein said polyamide layer comprises from 0 to 20% of at least one resin selected from the group, comprising polyesters, olefin homopolymers, olefin copolymers, ionomers, or their blend.
8. The synthetic casing according to claim 1, wherein said synthetic casing is made in a form of tube.

9. The synthetic casing according to claim 1, wherein said synthetic casing is made in a form of half-tube or sheet.

10. The synthetic casing according to claim 1, wherein said synthetic casing additionally comprises from 1 to 11 thermoplastic resin layers.

5 11. The synthetic casing according to claim 10, wherein said thermoplastic resin are selected from the group, comprising (co)polyamides, olefin (co)polymers, maleic anhydride grafted olefin (co)polymers, polyvinylidene chloride, ethylene/vinyl alcohol copolymers, thermoplastic polyurethanes, and also variant blends of above resins, belonging both to the same type and to different types.

10 12. The synthetic casing according to claim 11, wherein said (co)polyamides are polyamide 6, polyamide 66, copolyamide 6/66, polyamide 9, polyamide 10, polyamide 610, polyamide 612, copolyamide 6/66/10, copolyamide 6/66/12, copolyamide 6I/6T, polyamide MXD6.

15 13. The synthetic casing according to claim 11, wherein said olefin (co)polymers are polypropylene, propylene/ethylene copolymers, comprising at least 80 mole % of propylene monomeric units, low density polyethylene prepared by radical polymerization, high density polyethylene and moderate density polyethylene prepared using Ziegler-Natta catalysts, linear low density and very low density polyethylenes prepared using metallocene catalysts, ethylene/vinyl acetate copolymers, ethylene/(meth) acrylic acid  
20 copolymers and ionomers.

14. The synthetic casing according to claim 11, wherein said ethylene/vinyl alcohol copolymers comprise from 32 to 44 mole % ethylene monomeric units.

25 15. The synthetic casing according to claim 11, wherein said polyvinylidene chloride is a copolymer of vinylidene chloride and at least one comonomer, selected from the group, comprising vinyl chloride and vinyl acetate, wherein mole percentage of vinylidene chloride is between 75 and 90 mole %.

30 16. The synthetic casing according to claim 1, wherein any of its layers additionally comprises from 0 to 10 wt. % based on total weight of the layer of composition of one of low molecular weight additives; selected from the group, comprising colorants, pigments, fillers, lubricants, processing aids, bubbling agents, slipping agents, matting agents.

17. The synthetic casing according to claim 8, wherein said synthetic casing has an internal food contact layer, preferably consisting of (co)polyamide or linear polyurethane, and in the most preferable case consisting of (co)polyamide.

18. The synthetic casing according to claim 9, wherein said synthetic casing has either only food contact layer or both said food contact layer and opposite side external layer, that are made of heat-sealable resin.

19. The synthetic casing according to claim 18, wherein said heat-sealable resin  
5 comprises at least one heat-sealable polymer having melting point in a range between 101 and 160°C and selected from the group, comprising metallocene low density polyethylene, metallocene very low density polyethylene, ethylene/vinyl acetate copolymer, ethylene/(meth)acrylic acid copolymers and ionomers, polyvinylidene chloride, copolyamide 6/66/12 or their blends.

10 20. The synthetic casing according to claim 1, wherein said at least one polyamide layer of the synthetic casing is located between two moisture-barrier layers.

21. The synthetic casing according to claim 20, wherein said moisture-barrier layers comprise at least one thermoplastic resin, selected from the group, comprising polypropylene, propylene/ethylene copolymers, comprising at least 80 mole % of  
15 propylene monomeric units, low density polyethylene prepared by radical polymerization, high density polyethylene and moderate density polyethylene prepared using Ziegler-Natta catalysts, linear low density and very low density polyethylenes prepared using metallocene catalysts, ethylene/vinyl acetate copolymers, and also variant blend of above resins.

20 22. The synthetic casing according to claim 10, wherein its layer made of resins of different nature and having scarcely or no inherent mutual adhesion are bound by adhesive layers made of maleic anhydride grafted olefin (co)polymers.

23. The synthetic casing according to claim 1, wherein the total thickness of its polyamide layers is in a range between 40 and 100% from wherein the total thickness of  
25 said casing and preferably between 45 and 100%.

24. The synthetic casing according to claim 1, wherein said synthetic casing has the total thickness from 18 to 120  $\mu\text{m}$ .

25. The synthetic casing according to claim 2, wherein said synthetic casing is made in form of shirred stick.

30 26. The synthetic casing according to claim 1, wherein said synthetic casing has an applied printed image.

27. A method for production of the synthetic casing according to any claims from 1 to 26, comprising consecutive steps of single- or multilayer extrudate extrusion, resulting in preparing of a primary tube; orientation draw of said primary tube, resulting in

preparing of a oriented tubular film; thermofixation of said oriented tubular film resulting in preparing of an oriented thermofixed tubular film, wherein said oriented thermofixed tubular film is further subjected to an additional treatment where said oriented thermofixed tubular film having temperature 60-140°C is drawn by 3-8% in the machine  
5 direction and 4-10% in the transverse direction, then cooled, while it is in the strained state, to the temperature that is not higher than glass transition temperature of the polyamide composition of the polyamide layer and wound up on a core.

28. The method according to claim 27 wherein during said additional treatment the oriented thermofixed tubular film is drawn in 4-8% in the machine direction and 5-10% in  
10 the transverse direction.

29. The method according to claim 27 wherein said thermofixation is carried out by heating of the oriented tubular film, being inflated by air in bubble form, to 130-160°C for 3-5 seconds.

30. The method according to claim 29 wherein said thermofixation is accompanied  
15 by shrinkage of no more than 10%, preferably 5-10% both in the machine and transverse direction.

31. The method according to claim 30 wherein said drawing during said additional treatment is performed at 70-130°C.

32. The method according to claim 31 wherein said cooling during said additional  
20 treatment is performed to the temperature not higher than temperature ranging between 45 and 57°C, depending on the composition of the polyamide layer.

33. The method according to claim 27 wherein said drawing during said additional treatment is performed by means of air with excessive pressure, blown into said tubular film, and two pairs of nip rollers, revolving with different speeds.

25 34. The method according to claim 27 wherein said additional treatment of the oriented tubular film is performed in the same manufacturing line as steps for production of the oriented tubular film.

35. The method according to claim 27 wherein said additional treatment of the oriented tubular film is performed off-line in a separate apparatus.

30 36. The method according to claim 35 wherein said additional treatment of the oriented tubular film is performed after application of a printed image onto said oriented tubular film.

37. The method according to claim 35 or 36 wherein said additional treatment of the oriented tubular film is performed in a separate apparatus with its further shirring and said separate apparatus is aggregated with a shirring machine in one manufacturing line.

38. The method according to claim 27 wherein said ready to be filled casing synthetic  
5 heat-shrinkable sausage casing is cut along one or both bent lines of flattened tube resulting in making of correspondently half-tube or extensive sheet.

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