

UNITED STATES PATENT OFFICE

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PRODUCTION OF ARTIFICIAL FILAMENTS
OR THREADS

Reginald Henry John Riley, Reginald Henry
Parkinson, and Herbert Henry Sims, Spondon,
near Derby, England, assignors to Celanese
Corporation of America, a corporation of Dela-
ware

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This invention relates to the production of arti-
ficial filaments, threads, yarns, ribbons, fabrics,
films or other materials having a basis of cellu-
lose acetate or of other organic esters or ethers
of cellulose and having a reduced, subdued or
modified lustre.

The manufacture of artificial filaments and
other products having a subdued lustre has re-
cently become of great importance. It is well-
known that the normal lustre of artificial silk is
very much higher than that of natural silk, and
the primary object of the present invention is
to produce filaments and other products having
a lustre below that of the normal high lustre
and preferably approximating to that of natural
silk or even lower. In the manufacture of viscose
filaments subdued lustre has been produced by
an addition to the spinning solution of petroleum
and like hydrocarbons, but this method is in-
applicable to the production of filaments of sub-
dued lustre from organic derivatives of cellulose.
It is possible to obtain filaments of subdued lustre
with cellulose acetate and like cellulose deriva-
tives by incorporating in the spinning solution
insoluble inorganic substances. There is no dif-
ficulty in reducing the particles of inorganic mat-
ter to the requisite state of subdivision to enable
them to pass the spinning nozzles and/or filters
used in the manufacture of artificial silk. The
incorporation of such inorganic substances in
sufficient quantity to produce an opacity in ac-
cordance with the commercial requirements leads
however to a somewhat serious reduction in the
strength of the filaments, while if inorganic sub-
stances be added to the spinning solution in a
quantity which does not materially reduce the
strength of the products the opacity of the fila-
ments and other products is not sufficient for
commercial requirements.

We have now found that filaments, threads,
yarns, ribbons, films and other products having
a basis of cellulose acetate or other organic de-
rivative of cellulose may be produced having the
required subdued lustre and at the same time
without materially reducing the strength by in-
corporating in the spinning solution titanium
oxide alone or in admixture with other substances
or in the form of compounds. We find that ti-
tanium oxide is capable of being reduced to an
extremely fine state of division so that a suspen-
sion thereof in the spinning solution readily
passes the filters and/or spinning nozzles with-
out causing obstruction and at the same time
relatively small proportions are sufficient to pro-
duce the required opacity in the filaments or other

products. Thus for instance an addition to the
spinning solution of 0.5 to 1% of titanium oxide
on the weight of the cellulose acetate or other
cellulose derivative in the spinning solution is
sufficient to satisfy the commercial demands for
products of subdued lustre. The invention does
not however exclude the addition of larger pro-
portions of titanium oxide or mixtures or com-
pounds containing the same, for example propor-
tions of 2 to 4% or more on the weight of the
base.

We have found that titanium oxide is very
much more efficient in reducing the lustre than
other inorganic substances and that filaments
and other products having the required reduced
lustre may be readily produced without material
alteration of tenacity and other valuable prop-
erties, which accompanies the use of other inor-
ganic substances.

The incorporation of the titanium oxide or mix-
ture or compound containing the same into the
spinning solutions may be effected by any suitable
means. Highly satisfactory results are obtain-
able by working an aqueous paste of the titanium
oxide or the like with the solvent to be used for
making up the spinning solution, for example
acetone in the case of cellulose acetate. By this
means the acetone or other solvent may partial-
ly or entirely replace the water in the paste, and
the acetone-titanium oxide paste may then be
directly incorporated in the spinning solution.
Similarly good results may be obtained by work-
ing the aqueous paste with a small proportion of
the spinning solution and incorporating the
resulting suspension in the main body of spinning
solution. Alternatively an aqueous suspension or
dispersion containing titanium oxide or mixtures
or compounds thereof in the required state of
subdivision may be added directly to the spinning
solution or to the solvent before solution of the
cellulose acetate or other cellulose derivative.
Or again the titanium oxide or the like may be
incorporated for the cellulose acetate or other
cellulose derivative prior to making up the spin-
ning solution. Thus for instance the wet cellu-
lose acetate obtained by water precipitation of an
acetylation solution after ripening may be knead-
ed with finely divided titanium oxide until a
homogeneous distribution of the oxide through-
out the cellulose acetate is produced.

It is preferable to reduce the titanium oxide or
mixture or compound containing the same to the
required state of division prior to incorporation in
the spinning solution. To enable the titanium
oxide and the like to pass the spinning nozzles

and/or filters without causing obstruction it is sufficient to attain a particle size somewhat less than .003 millimetres, but in order to produce the maximum opacifying effect on the filaments or other products we prefer to reduce the titanium oxide to an average particle size of between .0001 and .00035 millimetres. This may be effected by grinding either dry or in the presence of liquids, for example water or in the solvent to be used for making up the spinning solution. To improve the state of dispersion the grinding may take place in the presence of dispersators and/or protective colloids, for example Turkey red oil (preferably applied in relatively small proportions) and/or in the presence of oils, such as oleic acid, linseed oil, olive oil or the like. The titanium oxide or the like may thus be brought to an aqueous or oily paste, and in this form be worked with the solvent for making up the spinning solution or with the spinning solution itself. In order to obtain as uniform a particle size as possible it is preferable to apply a preliminary dry grinding whether or not a subsequent wet grinding be employed. After grinding a suitable grading of the particles may be carried out, for example by means of a cyclone-separator or other means whereby the finer particles are carried off by a current of air or other gas with subsequent fractional settling from the gas current.

The titanium oxide or mixtures or compounds thereof should not be employed in the form in which it is precipitated directly from solutions of titanium salts, since the maximum opacity is not obtained by this means, and for this reason precipitation or decomposition of a soluble titanium salt within the filaments themselves is not effective and is not included within the scope of this invention. The titanium oxide alone or in admixture or in compound form should be subjected to a calcination after precipitation, for example a calcination at 600 to 1000° C. and preferably 700 to 900° C. The calcination may, if desired, be effected in presence of calcium phosphate or other agent adapted to inhibit colour change. For barium or calcium titanates, if the titanium oxide be used in compound form, calcination at about 1100° C. is very effective.

It is desirable that the titanium oxide should be as free as possible from acid. It may be thoroughly washed prior to incorporation in the spinning solution or other precautions may be taken to remove residual acidity or to prevent acidity from developing. Or alternatively substances capable of preventing acidity from developing may be incorporated therewith. The addition of zinc oxide or zinc carbonate is very effective for this purpose. Other additions may be made to the titanium oxide, for instance a mixed titanium oxide-barium sulphate may be used for the purpose of the present invention with or without zinc oxide, zinc carbonate or the like. Again an addition of boric acid or a borate, e. g., barium or calcium borate, in a proportion for instance of 2 to 6% on the weight of the titanium oxide tends to enhance still further the opacity of the products. Again if desired coloured substances, and especially inorganic coloured substances, may be added to the titanium oxide either to obtain special colour effects in the filaments or other products or for the purpose of correcting or slightly modifying the normal colour of the titanium oxide. Thus for instance dark blue or black coloured substances may be added in small proportions. The titanium oxide may be mixed with such further substances before or

after calcination, but preferably the whole mixture is calcined together. As a further alternative as already indicated the titanium oxide may be employed in the form of compounds for instance calcium or barium titanate.

Titanium oxide obtained by precipitation of titanium nitride or titanium cyano-nitride by means of sulphuric acid or other suitable reagent, especially when the precipitation is carried out in presence of a phosphate or phosphoric acid, is very finely divided, and moreover has a low specific gravity and is therefore valuable for use in the present processes. It may be calcined as described above.

The spinning solutions in which have been incorporated the titanium oxide may be spun by dry or wet spinning methods in the normal manner.

Novel effects may be produced by twisting, weaving, knitting or otherwise associating the filaments of modified lustre with the normal lustrous filaments of cellulose acetate or of other artificial silks or fibres or by associating the new filaments with other fibres, such as cotton, wool and the like. In addition novel yarns or threads containing both the filaments or the like of subdued or modified lustre and filaments or the like of normal lustre may be produced by twisting together dull filaments from one spinning nozzle with bright or normal lustrous filaments from another spinning nozzle, the twisting being effected continuously with production. Or again part of the jet orifices of a single nozzle may be fed with a spinning solution containing an insoluble substance and the remaining part of the jet orifices fed with normal spinning solution. The dull and bright filaments extruded from the single spinning jet may then be associated and/or twisted as desired. Fabrics prepared from such "mixed" yarns or threads exhibit important novel effects. Filaments of subdued lustre obtained according to this invention may be united to each other and/or to filaments of normal lustre so as to obtain special monofil threads, for example by the processes described in prior U. S. applications S. Nos. 393,286 filed September 17, 1929 and 393,287 filed September 17, 1929.

If desired the reduced, subdued or modified lustre effects of the present invention may be enhanced by applying the present processes in conjunction with the processes described in prior U. S. applications S. Nos. 413,500 filed December 12, 1929, 423,561 filed January 25, 1930 and 431,967 filed February 27, 1930 or other processes designed to modify the lustre of the artificial materials.

The invention has been described above more particularly with reference to the spinning of products having a basis of cellulose acetate. It may also be applied to the manufacture of products having a basis of other organic derivatives of cellulose, for instance cellulose formate, propionate or butyrate or methyl, ethyl or benzyl cellulose or mixed ether-esters.

What we claim and desire to secure by Letters Patent is:—

1. Process of producing yarns of differential luster, which comprises extruding a solution containing an organic derivative of cellulose and a finely divided pigment-like material, and a solution of an organic derivative of cellulose free from pigment, through different orifices in the same spinning jet, and associating the filaments so formed, continuously with their production, to form a composite yarn.

2. Process of producing yarns of differential luster, which comprises extruding a solution containing cellulose acetate and a finely divided pigment-like material, and a solution of cellulose acetate free from pigment, through different orifices in the same spinning jet, and associating the filaments so formed, continuously with their production, to form a composite yarn.

3. Process of producing yarns of differential luster, which comprises extruding a solution containing an organic derivative of cellulose and a finely divided pigment-like material, and a solution of an organic derivative of cellulose free from pigment, through different orifices in the same spinning jet, and treating the filaments so formed, continuously with their production, with a limited quantity of solvent to render the filaments tacky,

and causing the tacky filaments to adhere so as to form a single filament of larger cross section.

4. Process of producing yarns of differential luster, which comprises extruding a solution containing cellulose acetate and a finely divided pigment-like material, and a solution of cellulose acetate free from pigment, through different orifices in the same spinning jet, and treating the filaments so formed, continuously with their production, with a limited quantity of solvent to render the filaments tacky, and causing the tacky filaments to adhere, so as to form a single filament of larger cross-section.

REGINALD HENRY JOHN RILEY.
REGINALD HENRY PARKINSON.
HERBERT HENRY SIMS.