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FRICTION-ACTUATED EXTRUSION OF COPPER

This invention relates to the friction-actuated extrusion of copper.

By "friction-actuated" extrusion is meant a process
5 comprising the steps of feeding metal into one end of a
passageway formed between first and second members with
the second member having a greater surface area for
engaging the metal than the first member, the passageway
having an obstruction at the end remote from the end
10 into which the metal is fed and having at least one die
orifice associated with the obstructed end, moving the
passageway-defining surface of the second member
relative to the passageway-defining surface of the first
member in the direction towards the die orifice from the
15 first end to the obstructed end, frictional drag of the
passageway-defining surface of the second member drawing
the metal through the passageway and generating in it a
pressure that is sufficient to extrude it through the
die orifice. The obstructed end of the passageway may
20 be blocked substantially entirely, as described in
British Patent Specification No. 1370894 (United Kingdom
Atomic Energy Authority) but when, as in the most usual
practice (the Conform process) the passageway is arcuate
and the second member is a wheel with a groove formed in
25 its surface into which the first member projects and the
obstructed end is defined by an abutment projecting from
the first member, we prefer that the abutment member is

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of substantially smaller cross-section than the passageway so that it leaves a substantial gap between the abutment member and the groove surface. In this case metal can adhere to the groove surface, as
5 described in the specification of our granted UK Patent No. 2069398B, whereby a portion of the metal extrudes through the clearance and remains as a lining in the groove to re-enter the passageway at the entry end while the remainder of the metal extrudes through the
10 die orifice.

The Conform process was originally developed for the extrusion of metal rod in-feed. Etherington, in a paper entitled "Conform - a new concept for the continuous extrusion forming of metals" presented at the
15 Winter Annual Meeting in Detroit of the American Society of Mechanical Engineers, November 11-15 1973 (paper No. 73-WA/PT-2, reprinted by the United Kingdom Atomic Energy Authority as TRG Report 2530(S)) has proposed that the Conform process should be used to extrude
20 copper using an infeed of chopped copper cathodes. One of the steps during the preparation of the copper in-feed is the removal of oxide and other surface contaminations from the metal particles, because their presence can result in failure at the particle
25 boundaries (inter-particle separation) and/or blistering of the surface of the extruded copper product. In an analagous process using conventional ram extrusion

(Stout, American Institute of Mining and Metallurgical Engineers Technical Publication No. 1238, October 1940; Tyssowski American Institute of Mining and Metallurgical Engineers Paper No. 1217, February 1941.) a reducing
5 heat-treatment was applied after reduction to particulate form to remove oxide contamination. We have suggested (US-A-4110892) that such heat treatment is undesirable, and in consequence we have experimented by treating (pickling) granulated cathode copper with a
10 suitable chemical or chemicals, but we have found that pickling the granules will not, necessarily prevent blistering as contamination on the surface of the metal before granulating can become entrained into the metal during granulation, and will not therefore be removed by
15 pickling afterwards.

GB-A-698717 makes the unrealistic proposal that aluminium, or other metal of comparable susceptibility to oxidation, should be formed into small oxide-free pieces and fed as such in a vacuum or a protective
20 atmosphere to a screw extrusion press. It is said that the oxide might be removed, before or after the material is divided into pieces, by pickling, brushing or scalping. No suitable pickling solution is disclosed, and no merit is claimed for pickling before, rather than
25 after, division into pieces. The teaching of the document is, moreover, confined to "readily oxidisable metals" whereas copper is among the least reactive of metals that can be oxidised in the air.

In accordance with the invention, a process of friction actuated extrusion of copper comprises pickling cathode copper granulating the pickled copper and feeding the pickled granulated copper into a continuous friction-actuated extrusion machine without permitting significant oxidation or other film contamination after pickling.

After pickling the cathode is preferably washed in distilled or de-ionised water.

10 Preferably after pickling the copper is kept below 80°C, since above that temperature significant oxidation or other film contamination (e.g. sulphides) of the metal may occur. After pickling the metal is preferably stored in a protective (inert) atmosphere. Further, the
15 metal is preferably granulated in a protective (inert) atmosphere.

Using this arrangement it has been found that the previous disadvantage of blistering of the metal surface has been overcome.

20 The granulated metal may, additionally, be pickled before it is extruded.

Obviously any form of handling or storing of the cathode after pickling which is likely to lead to contamination (e.g. by grease, dust, dirt, etc) of the
25 cathode is preferably avoided.

Any suitable pickling solution may be used. The pickling solution preferably comprises a mixture of sulphuric acid, hydrogen peroxide and water. The

hydrogen peroxide provides extra oxygen to assist in the dissolution by the acid of copper (I) oxide by oxidation to copper (II) sulphate.

Other suitable pickling solutions include 10%
5 hydrochloric acid; 10% sulphuric acid; 25% sulphuric acid with 1.5% sodium bichromate; 50% sulphuric acid with 25% nitric acid; or 8% tartaric acid.

Example

A copper cathode was granulated in a granulator as
10 described in our published European Patent Application No. 94258 to an approximate size of 3 mm and used as the in-feed for a Conform machine as described in UK-B-2069398 using an extrusion ratio of approximately 20:1. The copper wire that was subsequently extruded blistered
15 during extrusion. Further, the wire that was extruded showed severe inter-particle separation during torsion, bend and tensile tests.

Another copper cathode was granulated as above,
pickled in 10% sulphuric acid and 3% hydrogen peroxide
20 at 40C for one hour, rinsed, washed, dried and then used as the in-feed for a Conform machine under the same conditions as above. The copper wire that was subsequently extruded blistered during extrusion, but showed no significant signs of inter-particle separation
25 during torsion, bend and tensile tests until after annealing for one hour at 400 to 500°C.

A further copper cathode was vapour degreased and then pickled in a dilute solution comprising 10%

suphuric acid and 3% hydrogen peroxide at 40-45°C for approximately 5 minutes. The cathode was then washed in de-ionised water and blow dried before being granulated and fed into a Conform machine under the same conditions as above. The copper wire that was extruded showed no signs of blistering or significant inter-particle separation, until after heating for one hour at 400 to 500°C. Some of the copper cathode was granulated in a nitrogen atmosphere and the resulting wire extruded under the same conditions withstood annealing at 600°C for one hour without showing signs of blistering or inter-particle separation. At all times during the preparation of the pickled cathode the temperature of the copper was kept below 80°C.

Using this invention, it is therefore possible to produce from granular cathode infeed which has not been heat treated extruded copper wire that is free from blistering.

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CLAIMS

1. A process of friction-actuated extrusion of copper comprising pickling cathode copper granulating the pickled copper and feeding the pickled, granulated
 - 5 copper into a continuous friction-actuated extrusion machine without permitting significant oxidation or other film contamination after pickling.
 2. A process as claimed in Claim 1, wherein after pickling, the copper is kept below 80°C.
 - 10 3. A process as claimed in Claim 1 or Claim 2 wherein the metal is granulated in a protective atmosphere.
 4. A process as claimed in any one of the preceding Claims, wherein the metal is additionally pickled after granulating.
 - 15 5. A process as claimed in any one of the preceding Claims in which after the cathode copper is pickled, it is washed in distilled or de-ionised water.
 6. A process as claimed in any one of the preceding Claims, wherein the pickling solution comprises a
 - 20 mixture of sulphuric acid, hydrogen peroxide and water.
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