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(54) METHOD FOR PRODUCING SHEET SEMIFINISHED PRODUCT FROM A TITANIUM ALLOY

(57) The invention relates to plastic working of metals, more specifically to rolling sheets, and is concerned with a method of manufacturing a semi-finished sheet product from a titanium alloy having a submicrocrystalline structure suitable for low-temperature superplastic deformation. The invention can be most efficiently used to manufacture semi-finished thin sheets, including foil, from a low-plastic two-phase titanium alloy.

The object of the invention is to improve quality of semi-finished sheet products made from a titanium alloy adapted for further low-temperature superplastic deformation.

A method of manufacturing a semi-finished sheet product from a titanium alloy adapted for low-temperature superplastic deformation, including rolling a billet with a prepared structure at a temperature below the polymorphous transformation temperature in isothermal or quasi-isothermal conditions provided by heating the rolls, the method **characterized in that** said rolling is carried out in conditions of low-temperature superplastic deformation, the deformation being performed, predominantly in a first pass, to a strain amount of $\varepsilon \geq \varepsilon_{min}$, where ε_{min} is the minimum amount at which a structural state required to provide cooperative grain boundary sliding in the deformation process is formed in the alloy in selected rolling temperature/rate conditions; after each subsequent rolling pass the billet is cooled immediately on exiting the deformation region to maintain the structural state obtained in the deformation; a time period of heating the billet in a furnace for a subsequent rolling pass is restricted to prevent disturbance of the alloy structural state obtained in the previous rolling pass.

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Description

[0001] The invention relates to plastic working of metals, more specifically to rolling sheets, and is concerned with a method of manufacturing a semi-finished sheet product from a titanium alloy having a submicrocrystalline structure suitable for low-temperature superplastic deformation. The invention can be most efficiently used to manufacture semi-finished thin sheets, including foil, from a low-plastic two-phase titanium alloy,

[0002] Quality of semi-finished sheet products is defined by the following characteristics that are interrelated through rolling schedules and methods: surface condition, accuracy of geometrical dimensions and shape, mechanical properties of semi-finished product defined by its structure, including grain size, and anisotropy or isotropy of mechanical properties provided by the type of metallographic texture formed in the process of rolling.

[0003] Tendency to improving the accuracy of semi-finished sheet dimensions and shape and the surface condition has been always essential. This is dictated, on one hand, by the effort to save metal, and, on the other hand, by toughened consumer demands associated with a specific character of some segments of industry. By way of example, a semi-finished sheet of uneven gauge cannot be subjected to drawing, the more so to deep drawing, since the deformation localization will inevitably cause irreversible defects across the wall thickness of articles to the point of fracture of the semi-finished sheet product in the drawing process. Sheets having rough surface and poor flatness are unsuitable for diffusion welding.

[0004] The tolerance range becomes yet narrower as the sheet thickness reduces.

[0005] Thickness of semi-finished sheet products can vary laterally and longitudinally, along and across the rolling direction, respectively. Disturbed shape generally manifests itself in waviness and camber of semi-finished sheets.

[0006] Any variation in rolling conditions and rolled metal characteristics affects the final thickness and shape of semi-finished sheet products. Rolling force is a major factor affecting formation and variation of the final thickness. It is assumed that elastic strains of a reduction mill is in direct proportion to the rolling force [1]. In hot and warm rolling, rolling force variations are also caused by changing the strain resistance of the material rolled, which is caused in turn by variation in rolling temperature. Furthermore, the rolling force depends on roll take-up and bounce, thickness of the billet rolled (rolled stock). Due to the process heredity existing in sheet rolling, variation of thickness appeared in a semi-finished sheet does not disappear completely despite the leveling effect of subsequent rolling processes and can be even aggravated at the rolling mills without control system.

[0007] In addition to the aforementioned, thickness and surface condition of a semi-finished sheet made of a titanium alloy depend to a great extent on the thickness of solid, gas-enriched and friable oxidized layer, scale, resulting from intense oxidation and saturation of the titanium billet surface with nitrogen and oxygen. Therefore, the manufacture of semi-finished sheet products having precise dimensions and clean surface by hot rolling is unfeasible without the use of vacuum or protective environment. But even with the use of vacuum or protective environment, elevated temperatures decrease rigidness of the roll system and increase elastic strain of the rolls. As the result, thin semi-finished sheets or foil cannot be generally produced by hot rolling.

[0008] Hot-rolled strips are generally used as semi-finished products for subsequent cold rolling.

[0009] Cold rolling of hot-rolled pickled strips is accomplished using intermediate annealing steps [1]. Low-plastic two-phase titanium alloys require a particularly great number of intermediate annealing steps. In the process of cold rolling the strain resistance increases, while the rolling forces and moments should not exceed the maximum allowed values for the rolling mill.

[0010] The cold-rolled semi-finished product has a pronounced metallographic texture leading to anisotropy of its properties. Anisotropy of semi-finished sheet product properties is undesirable in most cases. Electric steel is perhaps the only exception. If the semi-finished sheet product is to be further subjected to deformation to produce an article such as a shell, anisotropy of its properties is not only undesirable, but also inadmissible.

[0011] The problem can be tackled to a certain extent by the use of warm rolling at which the drawbacks inherent in hot and cold rolling are not so pronounced.

[0012] In addition, warm rolling provides the possibility to maintain, in a semi-finished sheet made from a two-phase titanium alloy, submicrocrystalline (SMC) or nanocrystalline (NC) structure, a fine-grain structure with a grain size of less than 1 μm and less than 0.1 μm, respectively, if the original billet exhibited such a structure. The necessity naturally arises to specially prepare such a structure in the original billet, but a variety of methods exist for this purpose. Currently, even a high-quality commercial rolled sheet has a grain size of about 3 to 5 μm. The presence of SMC and NC structure in a semi-finished sheet product is currently a pressing factor, as it will provide later on the fabrication of an intricately-shaped article by superplastic forming or by the combination of superplastic forming and diffusion welding (SPF/DW), using the low-temperature superplasticity effect. Reduction in the forming and/or diffusion-welding temperature ensures improved stability of the tool set used in the processes, and makes the processes more economical as a whole. Bonding of titanium semi-finished sheet products can be improved owing to reduced gas saturation of the surfaces to be joined. Furthermore, the SMC or NC structure of the semi-finished rolled product can be maintained in the ready article, this allowing the unique combination of mechanical properties inherent in SMC and NC materials and manifesting themselves in improved strength and fatigue characteristics to be implemented to the most extent. And at last, the unique possibility appears to accomplish the process of combined deformation of a billet made from aluminum or its alloy and a semi-finished sheet made from titanium alloy and to bind them by pressure welding at a temperature of about 400-450°C and lower at which no brittle intermetallic layer forms in the bond region. Articles in which such a bond can be implemented are of interest for current science and technology. By now, reference literature [2,3] mentions the possibility of binding at said temperatures a billet made from aluminum or its alloy and a billet made of only a sufficiently plastic, commercially pure titanium or low-alloy titanium alloys.

[0013] A method for manufacturing a semi-finished sheet product from a two-phase titanium alloy comprises warm rolling at a deformation starting temperature by 400-550°C lower than polymorphous transformation temperature with a strain rate of 10^{-4} to 10^{-2} s^{-1} and a strain amount from 5 to 15%, followed by annealing at a temperature by 400 to 550°C lower than the polymorphous transformation temperature, and repeating the treatment cycle to a total strain amount from 75 to 95% [4]. To prepare the structure, the billet is pre-treated in β and (α+β) regions prior to rolling. After the pre-treatment the billet has a coarse-grain lamellar or partly recrystallized globular/lamellar structure.

[0014] In the process of warm rolling under the aforementioned conditions, recrystallization and globularization processes take place. The processes do not however proceed uniformly and fully throughout the semi-finished sheet section. So, the resulting semi-finished product has a nonuniform microstructure, hence, nonuniform mechanical properties. The microstructure nonuniformity is inadmissible for further superplastic deformation. But even in the absence of microstructure nonuniformity, insufficiently fine grain size prevents the use of the resulting semi-finished product for low-temperature superplastic deformation.

[0015] Furthermore, variations in the rolling force caused by the structure nonuniformity bring about variations in the rolled billet thickness.

[0016] Warm rolling is carried out at DUO-200 rolling mill. Billets are heated in KS-300 electric resistance furnace.

[0017] A method of manufacturing a semi-finished sheet product from a titanium alloy comprises pre-treating a billet to a structure with submicron grain size, followed by rolling [5]. The rolling is started at a temperature in the range by 150-500°C below polymorphous transformation temperature dictated by the required submicron grain size in the semi-finished product to be produced. The resulting semi-finished sheet product is suitable for further treatment in low-temperature super-plasticity conditions. Although the rolling as such provides for the use of the temperature range including, as a component, the temperature range typical for low-temperature superplasticity, it is not performed in superplasticity conditions. Superplasticity conditions require strict correspondence of deformation parameters such as deformation temperature, strain rate and grain size in the billet being treated and observance of isothermal conditions in the deformation region.

[0018] In known method [5] the reduced temperature rolling to a desired thickness of the semi-finished product is conducted in several passes with partial reductions by 5-20%, and upon attaining the total strain amount of 40-65%, intermediate annealing is performed at a temperature below the polymorphous transformation temperature of the alloy by a value from 150 to 500°C.

[0019] The pre-treatment of the billet to a structure with submicron grain size is performed by plastic working methods different from rolling, as it is considered that intense and uniform plastic deformation of material across the billet section to create submicrocrystalline structure in a semi-finished sheet product merely by rolling is impossible. A method comprises multilateral deformation including a combination of swaging and drawing steps with changing of deformation axes.

[0020] In prior art method [5] rolling of the billet pretreated to a structure with submicron grain size is conducted within the range of existence of SMC structure in the material. Upper limit of the rolling temperature range is dictated by the desired submicron grain size in the resulting semi-finished sheet product. Lower limit is restricted by process plasticity of the SMC material being treated. Thus, in the rolling process either the original microstructure of the pre-treated billet is maintained, or grains are somewhat reduced or increased within the submicron range to the desired grain size in the resulting semi-finished product. As noted above, any change in the grain size causes variation in the plasticity of the material being rolled, the rolling force and, as consequence, variations in thickness of the semi-finished sheet product.

[0021] In the prior art method, a basic texture can be created to provide isotropy of mechanical properties in two directions in the sheet plane owing to the use of the longitudinal/transverse rolling step. The step can be however generally employed only for square sheets.

[0022] To reduce cooling-down the billet in the rolling process under non-isothermal conditions and to stabilize the rolling conditions the prior art method comprises heating the rolls. When said rolls are heated to a deformation temperature, the rolling is performed in isothermal conditions. The step is however optional in the method. Although, if the prior art rolling is started from a temperature lower than the polymorphic transformation temperature by 500°C, as the result of cooling-down at cold rolls, especially when thin semi-finished sheets are rolled, the rolling may become infeasible due to insufficient plasticity of the alloy.

[0023] Despite that fact that the grain size remains within the submicron range when temperatures in the range by 150-200°C below the polymorphous transformation temperature are used, oxygen and nitrogen, actively dissolve in titanium and form a gas-enriched layer and scale. To produce a semi-finished product with a predetermined thickness, either rolling should be carried out in vacuum, or the original billet should be protected by a coating. In the latter case, when making a thin semi-finished sheet or foil problems may arise in separating them from the coating, even up to crippling. In addition, the both steps are uneconomic. The economy is further impaired by the fact that SMC and even NC structure with a particular grain size of about a fraction of micrometer can be attained in a billet with the aid of rather labor-consuming steps, and this particular grain size can be then lost in the process of rolling at a temperature within the range by 150-200°C below polymorphous transformation temperature (T_{pt}).

[0024] The object of the present invention is to improve quality of semi-finished sheet products made from a titanium alloy adapted for further low-temperature superplastic deformation owing to stabilized grain size, more complete isotropy of properties, reduced variation in thickness of the semi-finished product and improved surface condition of said product, at reduced manufacturing costs of the semi-finished sheet product.

[0025] Another object of the present invention is to expand process capabilities of the method owing to production of especially thin semi-finished sheet products, including foil, with predetermined geometric dimensions, surface condition and grain size.

[0026] An object of the invention is to reduce potential variation in thickness of a semi-finished sheet product and improve its flatness.

[0027] A further object of the invention is to further reduce manufacturing costs of a semi-finished sheet product, including the step of preparing the structure in the original billet.

[0028] The objects of the invention are attained in a method of manufacturing a semi-finished sheet product from a titanium alloy adapted for low-temperature superplastic deformation, including rolling a billet with a prepared structure at a temperature below the polymorphous transformation temperature in isothermal or quasi-isothermal conditions provided by heating the rolls, wherein in accordance with the present invention said rolling is carried out in conditions of low-temperature superplastic deformation, the deformation being performed, predominantly in a first pass, to a strain amount of $\varepsilon \geq \varepsilon_{min}$, where ε_{min} is the minimum amount at which a structural state required to provide cooperative grain boundary sliding (CGBS) in the deformation is formed in the alloy in selected rolling temperature/rate conditions; after each subsequent rolling pass the billet is cooled immediately when exiting the deformation region to maintain the structural state obtained in the deformation; a time period of heating the billet in a furnace for a subsequent rolling pass is restricted to prevent disturbance of the alloy structural state obtained in the previous rolling pass.

[0029] The objects of the invention can be further attained by the following steps:

said rolling is carried out at a temperature in the range from T_{pt}-450°C to T_{pt}-350°C;
said rolling is carried out with a strain rate in the range from 10^{-3} to 10^{-1} s^{-1};
in said rolling, prior to achieving a strain amount of 30-60%, the billet is rotated through 90 degrees after every three to five longitudinal passes and a transverse rolling pass is performed, the remaining strain amount being gained by rolling in single direction;
when manufacturing a semi-finished sheet product having a thickness not exceeding 1 mm the billet is heated through contact with working rolls;
a billet with a prepared globular structure having a grain size less than 1 μm is used in said rolling;
a billet with a prepared lamellar structure having a cross-sectional grain size less than 1 μm is used in said rolling;
the billet structure is prepared for rolling by preliminarily rolling an original billet having a grain size not exceeding 10 μm at least in one section to a strain amount of at least 80%, said rolling being started at a temperature in the range from T_{pt}-300°C to T_{pt}-200°C and finished at a temperature not lower than the basic rolling temperature, wherein the strain rate is in the range from 10^{-2} to 10° s^{-1};
the billet structure is prepared for rolling by preliminary two-stage rolling of an original billet having a grain size of from 10 to 80 μm, the first stage comprising rolling the original billet to a strain amount not exceeding 60%, the rolling being started at a temperature in the range from T_{pt}-200° to T_{pt}-50° and finished at a temperature not lower than the basic rolling temperature, wherein the strain rate is in the range from 10^{-2} to 10 s^{-1}; the second stage comprising rolling the billet in isothermal conditions at the basic rolling temperature and strain rate to a strain amount of 20-30%;
said rolling is carried out at a rolling mill comprising two working rolls and at least four backup rolls;
deflection of the backup rolls directly contacting the working rolls is modified by changing the intensity of cooling bearings units of the backup rolls;
said working rolls are heated by electric resistance heating units mounted inside the rolls.

[0030] A principal distinctive feature of the invention is that the method is suitable not only for manufacturing semi-finished sheet products adapted for low-temperature superplastic deformation, but the rolling as such is carried out in conditions of low-temperature superplasticity. In this case the efforts directed at preparation of the billet structure for rolling are more strictly spent for intended purpose. But of more importance for attaining the objects of the invention is the change in the technical essence of the rolling method as compared to the prior art method.

[0031] To elucidate the technical essence of the invented method, consider physics of deformation process in superplasticity conditions in more details.

[0032] It is well known that deformation in superplasticity conditions proceeds to a considerable extent without accumulation of residual stresses and at smaller deforming forces, which is of special importance in processing low-plastic materials, including a two-phase titanium alloy.

[0033] The basic mechanism of superplastic deformation of an alloy is grain boundary slipping (GBS). Once a predetermined strain has been achieved, i.e. when all or a major part of the billet volume have been involved in the deformation process, GBS takes on a cooperative nature, CGBS [6, 7]. As compared to intragrain slip which is of secondary importance in deformation in superplasticity conditions, by GBS grains do not extend and remain equiaxial, or globular. As the result, formation of both metallographic and crystallographic texture takes place to a lesser extent. Moreover, if a texture existed in the original billet, it will dissipate in the deformation in superplasticity conditions owing to the CGBS. Therefore development of CGBS in the rolling process provides isotropy of semi-finished sheet properties in any arbitrary direction on the sheet plane.

[0034] Development of CGBS does not begin at once when a deforming force is applied. First, a shear band forms that unites a great number of series connected grain boundaries. This process proceeds on self-organization principle and is associated with increase in the angles in triple joints (rectification of boundaries). At this stage, corresponding to 3-15% deformation, the flow stress intensively grows, this leading to increased deforming force (Figs. 7,8). Once shear bands have been formed, the flow stress becomes steady or decreases gradually. A particular strain amount required to make the process steady depends on grain size in the billet being deformed; the less the grain size the smaller strain amount is required to form the desired structural state. The steady flow stage corresponds to superplasticity conditions where CGBS is the main deformation mechanism. Presence of formed shear bands, i.e. CGBS, is determined, as stated above, from the flow stress versus strain chart (hereinafter referred to as stress/strain) constructed for the most simple and illustrative case of applying a load to a specimen, uniaxial tension (continuous line in Fig.7). It should be noted that at small strain amounts (10-15%) values of relative tensile and rolling (in terms of reduction) strains are close and the comparison is rightful.

[0035] Such material behavior is however correct for monotonic deformation. In the rolling process, deformation has a fractionary nature. Deformation region is continuously displacing, rolling is accomplished in several passes, the one-pass strain amount being only 10-15% as noted above. Thus, it is important to form shear bands predominantly in the first pass, so that at the next passes, when a billet section appears again in the deformation region, the shear strips would be already formed and, as the result, plastic properties of the rolled material and rolling force would stabilize in every next pass, where possible.

[0036] Where a billet with a prepared globular SMC or NC structure is rolled, the structural state required to provide CGBS is achieved even after 5-7% strain. Where a billet with a prepared lamellar structure having plates of less than 1 μm cross-sectional size is rolled, the strain amount may be greater than in the previous case and reach 10-15%.

[0037] Another essential measure for rolling in superplasticity conditions is to cool the billet exiting the deformation region after the pass, this enabling the grain size and formed shear bands to be maintained, Holding the material between passes at a temperature close to the deformation temperature, which does not even leads to grain growth, gives rise to change in the grain boundary states and partial recovery of the original structure. All the more, annealing between passes leads to full recovery of equilibrium structure and coarsening the grains. In both cases, i.e. after holding and annealing, stresses increase as compared to stresses observed under continuous load for the same strains (Fig.7).

[0038] Fig. 8 shows plots of continuous and fractionary process of applying a load to a specimen with partial cooling (by 100°C) after removing the load, the plots noticeably approaching.

[0039] To maintain formed shear bands, a time period of heating the billet for next rolling pass is also restricted when furnace heating is used. The latter condition is not of necessity when a thin sheet stock is heated by contact with working rolls.

[0040] The novel, non-obvious measure comprising effecting plastic properties of the rolled material in order to stabilize them by maintaining the grain boundary conditions in the process of fractionary, non-monotonic rolling deformation is efficient just in warm rolling. In hot rolling this effect will be lost against the background of intense temperature effect on the roll system stiffness. This necessitates another measure, rolling in the conditions of low-temperature superplasticity.

[0041] Furthermore, warm rolling enables the manufacture of semi-finished sheet products with improved accuracy and surface condition owing to substantially complete exclusion of forming of a gas-enriched layer and scale. As a consequence, a wide range of semi-finished thin sheet products, including foils of different thickness, can be produced without the use of vacuum or protective environment. Reduced rolling forces, inherent in superplasticity, and elimination of further treatment of the semi-finished sheet product for removing the gas-enriched layer, as well as the absence of need to use vacuum substantially reduce the manufacturing costs of high-quality semi-finished sheet products despite the necessity to heat the rolls.

[0042] As noted above, the individual temperature range inherent in low-temperature superplasticity is a constituent part of the conventional temperature range. But this step is used in combination with the other steps involved in the method. The new combination provides numerous advantages. Thus, when considering the step of rolling in the temperature range which is a constituent part of the conventional range, as a component, it can be seen that the claimed technical solution provides a super-cumulative effect as compared to the prior art solutions.

[0043] To observe the low-temperature superplasticity conditions as a whole, the alloy should have a homogeneous, equiaxial fine-grain structure, and deformation should be carried out in isothermal conditions.

[0044] Each particular temperature of deformation carried out in superplasticity conditions is associated with a specific grain size. For low-temperature superplasticity the grain size is less than 1 μm. As current commercial rolled products fail to observe this requirement, the billet structure is to be specially prepared.

[0045] In the rolling process, isothermal conditions imply constant temperature in the deformation region. The step of heating the rolls in the invented method is therefore essential for attaining the objects of the invention. Mechanical heating caused by the deformation, process occurring at a rate inherent in low-temperature superplastic deformation may be neglected. Alternatively, mechanical heating of the billet can be fully compensated by the choice of a corresponding, lower roll temperature. By way of example, if at a strain rate $\varepsilon'=10^{-2}s^{-1}$ the billet is heated by 30°C for the rolling time, then isothermal conditions will be strictly observed in the deformation region at the roll temperature of 470°C as compared to the required temperature of 500°C.

[0046] It should be noted that where thin sheets are rolled a greater total strain amount is gained, and in combination with low-temperature superplasticity conditions this influences the final structure, namely, causes additional grain refining, which can be considered as an advantage of the method.

[0047] Thus, attainment of the aforementioned objects is provided by the entire combination of features of the claimed invention.

[0048] The technical matter of the invention will be further described in more details.

[0049] Described optimal deformation temperature and rate ranges in low-temperature superplasticity conditions were experimentally tested and found suitable for the most of titanium alloys.

[0050] A step of rotating, when being rolled, a billet through 90° after every three to five passes until a strain amount of 30-60% is attained, and carrying out a transverse pass, while the remaining part of strain is gained by rolling in single direction, improves flatness of the rolled sheet. Once 60% reduction has been attained, the flatness distortion is of no importance. In the prior art method [5] a similar step is used only to provide anisotropy of properties of the rolled sheet in respective directions on its plane.

[0051] When manufacturing a semi-finished sheet product having a thickness of no more than 1 mm, a billet is heated immediately by contact with the working rolls. The process can be considered as quasi-isothermal. Owing to small sheet thickness and low rate of rolling, the required temperature is sufficiently fast established in the deformation region, even at the initial rolling step. A billet of a greater thickness is heated slowly or even have no time to heat to a predetermined temperature for the rolling time, thus such a billet should be heated in a furnace immediately before the rolling. A through-type furnace is generally used in this case.

[0052] To provide low-temperature superplasticity and CGBS a billet with a prepared structure is used in the rolling process. The structure must be a homogeneous structure with equiaxial (globular) grains of less than 1 μm size. This structure may be provided in the original billet, i.e. using known methods [4,5]. In this case to make CGBS "wok" it is sufficient to only form shear bands between the grains, this corresponding to about 5-10% strain as mentioned above.

[0053] The structure can be alternatively prepared so that to transform to the required structure in the rolling process, preferably in a first pass. A lamellar structure with elongated grains having a cross-sectional size from 0.9 to 1.5 μm meets this requirement. Low rolling temperature and strain rate provide dynamic recrystallization process with division of plates and formation of fine, about 0.2 μm, equiaxial grains. Isothermal conditions provide uniform behavior of the process and its smooth transformation to shear band formation process. This requires approximately 10-15% strain. In the following process, when CGBS is developing, i.e. in the absence of dynamic recrystallization, grains will retain their shape and size.

[0054] Such a lamellar structure can be provided by preliminarily rolling the original billet.

[0055] If the original billet grain size does not exceed 10 μm, the billet is subjected to preliminary rolling started at a temperature below the polymorphous transformation temperature by 200-300°C and finished at a temperature not less than the basic rolling temperature at a strain rate in the range from 10^{-2} to $10°\ s^{-1}$, The strain rate in this range promotes active dynamic recrystallization process. The strain amount exceeds the amount required to develop dynamic recrystallization, about 70%. The latter means that in the deformation process grains acquire equiaxial shape, and then lost it again, i.e. become elongated. The resulting billet acquires a lamellar structure with plates having a cross-sectional size of 0.9 to 1.5 μm.

[0056] If the grain size in the original billet exceeds 10 μm, the billet is subjected to two-stage preliminary rolling. At a first stage, the original billet is rolled to a strain amount not exceeding 60%, the rolling being started in the temperature range from T_{pt}-300°C to T_{pt}-200°C and finished at a temperature not less than the rolling temperature at a strain rate in the range from 10^{-2} to $10\ s^{-1}$. At the second stage, the billet is rolled in isothermal conditions at the basic rolling temperature and strain rate until a strain amount of 20-30% is attained. A feature of the two-stage rolling is that the strain amount at the first stage must be smaller than the strain amount causing formation of equiaxial (globular) grains in the billet owing to dynamic recrystallization. Deformation to a strain amount less than 60% causes only extension and thinning of the plates. If the grains become equiaxial and comparatively coarse, substantial deformation will be further required to render them plate-shaped. Thin plates may be obtained if development of dynamic recrystallization process is excluded. Then, by deforming thin plates at a lower temperature, precisely at the basic rolling temperature, finer plates can be obtained having a desired cross-sectional size. Similarly to the previous case, but only at the second stage of preliminary rolling, when the total strain amount of about 70% has been reached, the dynamic recrystallization process takes place with formation of equiaxial grains. The formed grains are reasonably fine owing to a lower deformation temperature. As the deformation continues, the grains become elongate again. As the result, as in the previous case, the billet acquires a lamellar structure with plates of less than 1 μm in cross section.

[0057] In both cases heating for the first pass may be accompanied by static recrystallization that promotes some conditioning of the structure and globularization of the grains. The structure will be completely conditioned and grains will acquire equiaxial shape even in the basic rolling process.

[0058] The most precise dimensions of semi-finished sheet product can be obtained if the rolling is carried out at a rolling mill comprising two working rolls and at least four backup rolls, e.g. at a six roll mill (Fig.1).

[0059] Cooling of backup rolls that directly contact the working rolls enables, first, the roll system rigidity to be increased. Second, non-uniform cooling of the backup rolls directly contacting the working rolls provides a slight gradient along the working roll body that is sufficient to reduce lateral variation in the sheet thickness. Backup rolls can be most optimally cooled by cooling respective bearing units. Intensity of cooling the bearing units depends on the required roll body size.

[0060] To heat the working rolls it is recommended to use a controlled electric resistance heating unit built-in in the roll, this allowing the optimal roll temperature to be set on the basis of the alloy grade and the grain size in the billet to be rolled.

[0061] The present invention will be further illustrated with the aid of the accompanying drawings, wherein:

Fig.1 shows a schematic diagram of the method;
Fig.2 shows the microstructure (a) and electron diffraction pattern (b) of original billet;
Fig.3 shows the microstructure and electron diffraction pattern of a semi-finished sheet product obtained after rolling the original billet with a structure prepared by methods other than rolling;
Fig.4 shows the microstructure of the original billet prepared by one stage rolling, x500;
Fig.5 shows the microstructure of the original billet prepared by two-stage rolling, x500;
Fig.6 shows the microstructure of semi-finished sheet product produced after rolling the original billet with a structure prepared by rolling;
Fig.7 shows the stress/strain plots for continuous and fractionary process with intermediate non-loaded annealing for 1 min;
Fig.8 shows stress/strain plots for continuous and fractionary process with partial cooling (by 100° at load removed;
Fig.9 shows the estimated deflection of a backup roll under experimentally found rolling force. Maximum deflection difference between the body center and end is 0.054 mm. It can be compensated by a temperature difference of 40°C at TEC 18×10^{-6} and roll diameter of 150 mm.

[0062] Fig.1 shows a billet 1 to be rolled, working rolls 2 with built-in heating units (not shown), backup rolls 3 (four), pre-heating through-type furnace 4.

Examples of Implementing the Method

[0063] It is to be understood that the examples of implementing the method will not limit the present invention in terms of titanium alloys that can be employed and dimensions of semi-finished sheet products.

[0064] Examples describe methods of manufacturing a semi-finished sheet 0.3 mm thick and foil 0.05 mm thick from BT-6 and BT-22 titanium alloys.

[0065] Two-phase titanium alloys, But22 and BT6, were processed.

[0066] Table 1 shows polymorphous transformation temperatures and chemical compositions, in percent by weight. In the Examples strips having a thickness of 0.1; 0.5 and 0.7 mm were manufactured.

Table 1

Alloy	T_{pt}	Ti	Al	V	Mo	Cr	Fe	Other
BT22	860°C	83. 8	4.48	4.29	4.62	0.95	0.98	0.88
BT6	980°C	89. 3	6.1	4.1	≤0.1	≤0.1	0.15	0.18

[0067] Example 1. A rolled sheet 0.5 mm thick was manufactured from a two-phase BT6 titanium alloy. An original billet with a thickness of 14 mm and a size of 60 $\times$ 100 mm having the grain size of 0.4 μm (Fig.2) was made by multiaxis swaging at a temperature reduced to 600°C [5].

[0068] Rolling was performed at a temperature of 560°C, which is by 430°C lower than polymorphous transformation temperature. Peripheral roll speed was 1 mm/s, which corresponded to a strain rate of 5 $\times$ 10^{-3} s^{-1} in the deformation region.

[0069] Prior to rolling, tensile specimens were cut out from the original billet to determine a minimum strain amount (ε_{min}) at which the alloy structural state required to provide CGBS in the deformation process is attained under the selected rolling temperature/rate conditions. ε_{min}=9% was determined on the basis of the maximum flow stress value after which it gradually reduces (Fig.8).

[0070] The original billet was rolled at LIS-6/200 six-roll mill comprising heated working rolls of 65 mm in diameter (Fig.1). The working rolls were heated to 560°C. Built-in resistance heating units heated the rolls on the inside. Backup rolls were heated by contact with the working rolls, the temperature attaining 120-180°C in the roll body center. The backup rolls were cooled a liquid lubricant circulating through bearing units. Intensity of the cooling was selected to provide a temperature difference of 40 $\pm$ 5°C between the center and ends of the backup roll bodies, this compensating deflection of the rolls and providing uniform thickness of sheet. Temperature of the backup rolls was controlled by a temperature control unit. A through-type furnace with a heating temperature of 560°C was provided at the mill input (Fig. 1). Strain amount in the first pass was 15%. As the final thickness approached, the one-pass strain amount was reduced. The total number of passes was 32.

[0071] The billet was air cooled to a temperature of 400-450°C after each pass at output of the deformation region. Before starting each next pass the billet was placed in the through-type furnace. Time period of heating was determined on the basis of one minute per one mm of thickness, which was sufficient only to heat the billet and did dot not assume holding the billet at the temperature. As the strip reached the thickness of 5 mm, the billet length exceeded the length of the through-type furnace. Then, the billet head was placed in the through-type furnace prior to rolling and heated for the time period of t=0.9h min, where h is the billet thickness. The billet was then supplied to the rolls. The remaining part of the billet was heated by the through-type furnace as the strip entered the deformation region. To provide heating of the billet, the length of heated zone in the furnace was estimated as $l \geq 54 \nu \cdot h$, where ν is the peripheral roll speed. Thus, at ν=1 mm/s and the billet thickness of h=5 mm the heated zone length should be at least 250 mm. At the same time, a long billet could not be fully heated since at a low rolling rate this would lead to holding at the rolling temperature. In this example the heated zone length in the furnace was 300 mm. This measure provided heating, but restricted the billet residence time under the rolling temperature, so the material structural state required to implement the basic superplasticity mechanism was maintained between passes.

[0072] As the strip thickness achieved 2 mm, the furnace temperature was set to 400-450°C to avoid annealing before supplying to the rolls, and final heating was performed directly by the working rolls when the strip entered the contact zone.

[0073] The resulting sheets were thoroughly examined. Variation in sheet thickness from the specified thickness of 0.5 mm did not exceed 0.02 mm. The surface was covered with a dark-blue, dense, thin oxide film without signs of scale. Microstructural analysis and microhardness measurements did not reveal a gas-enriched surface layer at a depth of more than 1 μm. The strip had a homogeneous globular microstructure with grains 0.2 μm in size and the elongation factor not exceeding 1.45 (Fig.3), while the total strip elongation was e=13. Intensity of texture maximums defining the amount of anisotropy did not exceed two pole density units.

[0074] Example 2. An original billet of 15 mm in thickness and 60x80 mm in size was made from a two-phase BT22 titanium alloy with a grain size of 0.6 μm by multiaxis swaging at reduced temperature [5]. A tensile specimen was cut out from the original billet at electrospark discharge machine to determine a minimum strain amount required to attain stable superplastic flow at a predetermined temperature, ε_{min}=11%. The billet was rolled to a thickness of 0.7 mm at LIS-6/200 six-roll mill comprising heated working rolls of 65 mm in diameter. The roll heating temperature was 550°C, which was by 310° lower than polymorphous transformation temperature. A through-type furnace at the mill input provided a heating temperature of 550°C. Peripheral roll speed was 1 mm/s, which provided a strain rate of 6 $\times$ 10^{-3} s^{-1} in the deformation region at 10% one-pass strain. This corresponded to the conditions of low-temperature superplasticity for given alloy. Usage of the through-type furnace eliminated cooling the billet at the so small supply speed. At output of the deformation region the billet was air cooled. The resulting strips were covered with a dark-blue, dense, thin, oxide film. Microstructural analysis and microhardness measurements did not reveal a gas-enriched surface layer at least at a depth of more than 1 μm. Variation in sheet thickness from the specified thickness of 0.7 mm did not exceed 0.01 mm. The strip had a homogeneous microstructure in cross section with grains 0.3 μm in size, and the elongation factor not exceeding 1.4. The total strip elongation factor was e=20.4. X-ray analysis did not reveal any signs of intense crystallographic texture.

[0075] Example 3. The procedure was similar to that described in Example 1 except that the rolling temperature was 600°C, and the one-pass strain was 20% at the initial stage. At the same peripheral roll speed (1 mm/s) the strain rate in the deformation region was 1.1 $\times$ $10^{-2} s^{-1}$. This also corresponded to low-temperature superplasticity conditions for the alloy with given grain size at this temperature. As the result, the number of passes was reduced to 23 while geometry of the produced sheet was maintained. It this case the grain size of the original billet was maintained. This measure substantially improved the process efficiency. Temperature in the through-type furnace was 580°C, taking into account the initial deformation heating.

[0076] Example 4. The procedure was similar to that described in Example 1 except that at the initial rolling stage the billet was rotated through 90° after every three passes and rolled in transverse direction. This step was performed until 60% strain was attained, in this case the billet width reached the width of the roll body (200 mm). To provide a desired combination of strain amount and billet width, the original billet size was 16x60x80 mm in contrast to that in Example 1. This measure provided the following results:

1) billet flatness was improved, providing thereby more uniform strain in subsequent passes;
2) width of the rolled billet was increased if the original billet had restricted dimensions, e.g. a conventional rod shape;
3) grain elongation factor was increased to 1.2 (in the sheet plane) and intensity of texture maximums was reduced.

[0077] Further rolling was carried out in single direction until desired strip dimensions were attained.

[0078] Example 5. The procedure was similar to that described in Example 1, but the aim was to manufacture sheets less then 0.5 mm thick. Once this value was attained, the strip was fed to hot rolls without pre-heating, or the temperature of input device was set to a value not exceeding half of the rolling temperature. This step restricted to the limit the billet residence time at the rolling temperature, ensuring thereby maintenance of the material superplasticity state between passes and providing more precise width of the resulting rolled product. To this end, the heating power of the working rolls should be raised to some extent to compensate heat loss for heating the billet. If the roll heating units have a heating power margin and comprise a feedback heat controller, the heating power will be automatically compensated. Automatic operation of the heating units requires about 30% power margin in excess of the rated value. The resulting foil specimens had a thickness of 0.1±.0.01 mm.

[0079] Example 6. A commercial rod of 60 mm in diameter from BT22 alloy with lamellar structure comprising plates having the average size of 80x6 μm was used as an original billet. The billet was heated to a temperature of 850°C, which was by 30°C lower than polymorphic transformation temperature and by 300°C higher than the basic rolling temperature. The billet was rolled at DUO 300 rolling mill using cold rolls with a rate of 200 mm/s. At 20% one-pass strain this corresponded to the strain rate of 1.2 s^{-1} in the deformation region. Rolling was performed in several passes to a thickness of 10 mm, which corresponded to 83% reduction. After first three passes the billet was rotated and rolled transversely in one pass. In the preliminary rolling the billet temperature was decreased by 10-15°C in each pass until it reached 700°C. With the temperature decrease the one-pass strain amount was reduced, such that the strain rate gradually reduced to values below 10 s^{-1} in the deformation region. Then scale and gas-enriched layer 0.12 mm thick were removed from each side of the billet. The resulting lamellar structure had thin grains elongated in the rolling direction with the mean size of 1.3 μm in lateral direction (Fig.4). Final rolling was performed in the way similar to that described in Example 2. However, the rolling stand pressure force had to be increased at the first pass. The lamellar structure was then gradually transforming to globular submicrocrystalline structure, and the process changed to the low-temperature superplasticity regime. The resulting sheet had a less homogenous structure than the submicrocrystalline billet, although the structure was still submicrocrystalline with grains of 0.4-0.5μm in size and the elongation factor of 1.4 in the strip longitudinal section. Crystallographic texture was feebly marked.

[0080] Example 7. An original billet was a billet of 100x60x60 mm in size made from BT22 alloy with the average grain size of 50 μm. The billet was heated to 820°C. Then the billet was rolled at DUO 300 rolling mill using cold rolls with a rate of 100 mm/s. At 20% one-pass strain this corresponded to a strain rate of 0.7 s^{-1} in the deformation region. Rolling was performed in two stages, each stage including several passes. The first stage included rolling to a thickness of 27 mm which corresponded to 55% reduction. In the preliminary rolling the billet temperature was decreased by 10-15°C in each pass until the temperature of 650° was reached. As temperature decreased, the one-pass strain amount was reduced, which resulted in gradually reduced strain rate to values below $10^{-1}s^{-1}$ in the deformation region. Then scale and gas-enriched layer of 0.12 mm thick were removed from each side of the billet. The resulting elongated grains had the average cross-sectional size of 1.9 μm. At the second stage, rolling was carried out in isothermal conditions at a temperature of 550°C in several passes to attain the total strain amount of 28%. The resulting structure is shown in Fig. 5. The average cross-sectional grain size was 0.9 μm. Basic rolling was performed in a manner similar to that described in Example 6.

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[0081]

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Claims

1. A method of manufacturing a semi-finished sheet product from a titanium alloy adapted for low-temperature superplastic deformation, including rolling a billet with a prepared structure at a temperature below the polymorphous transformation temperature in isothermal or quasi-isothermal conditions provided by heating the rolls,
the method **characterized in that**
said rolling is carried out in conditions of low-temperature superplastic deformation, the deformation being performed, preferably in a first pass, to a strain amount of $\varepsilon \geq \varepsilon_{min}$, where ε_{min} is the minimum amount at which a structural state required to provide cooperative grain boundary sliding in the deformation is formed in the alloy in selected rolling temperature/rate conditions;
after each subsequent rolling pass the billet is cooled immediately on exiting the deformation region to maintain the structural state obtained in the deformation process;
a time period of heating the billet in a furnace for a subsequent rolling pass is restricted to prevent disturbance of the alloy structural state obtained in the previous rolling pass.

2. The method according to claim 1, wherein said rolling is carried out at a temperature in the range from T_{pt}-450°C to T_{pt}-350°C.

3. The method according to claim 1, wherein said rolling is carried out with a strain rate in the range from 10^{-3} to 10^{-1} s^{-1}.

4. The method according to claim 1, wherein in said rolling, prior to achieving a strain amount of 30-60%, the billet is rotated through 90 degrees after every three to five longitudinal passes and a transverse rolling pass is performed, the remaining strain amount being gained by rolling in single direction.

5. The method according to claim 1, wherein when manufacturing a semi-finished sheet product having a thickness not exceeding 1 mm the billet is heated through contact with working rolls.

6. The method according to claim 1, wherein a billet with a prepared globular structure having a grain size less than 1 μm is used in said rolling.

7. The method according to claim 1, wherein a billet with a prepared lamellar structure having a cross-sectional grain size about 1 μm is used in said rolling.

8. The method according to claim 7, wherein the billet structure is prepared for rolling by preliminarily rolling an original billet having a grain size not exceeding 10 μm at least in one section to a strain amount of at least 80%, said rolling being started at a temperature in the range from T_{pt}-300°C to T_{pt}-200°C and finished at a temperature not lower than the basic rolling temperature, wherein the strain rate is in the range from 10^{-2} to 10° s^{-1}.

9. The method according to claim 7, wherein the billet structure is prepared for rolling by preliminary two-stage rolling of an original billet having a grain size of from 10 to 80 μm, the first stage comprising rolling the original billet to a strain amount not exceeding 60%, the rolling being started at a temperature in the range from T_{pt}-200° to .T_{pt}-50° and finished at a temperature not lower than the basic rolling temperature, wherein the strain rate is in the range from 10^{-2} to 10 s^{-1}; the second stage comprising rolling the billet in isothermal conditions at the basic rolling temperature and strain rate to a strain amount of 20-30%.

10. The method according to claim 1, wherein said rolling is carried out at a rolling mill comprising two working rolls and at least four backup rolls.

11. The method according to claim 10, wherein deflection of the backup rolls directly contacting the working rolls is modified by changing the intensity of cooling bearings units of the backup rolls.

12. The method according to claim 1, wherein said working rolls are heated by electric resistance heating units mounted inside the rolls.

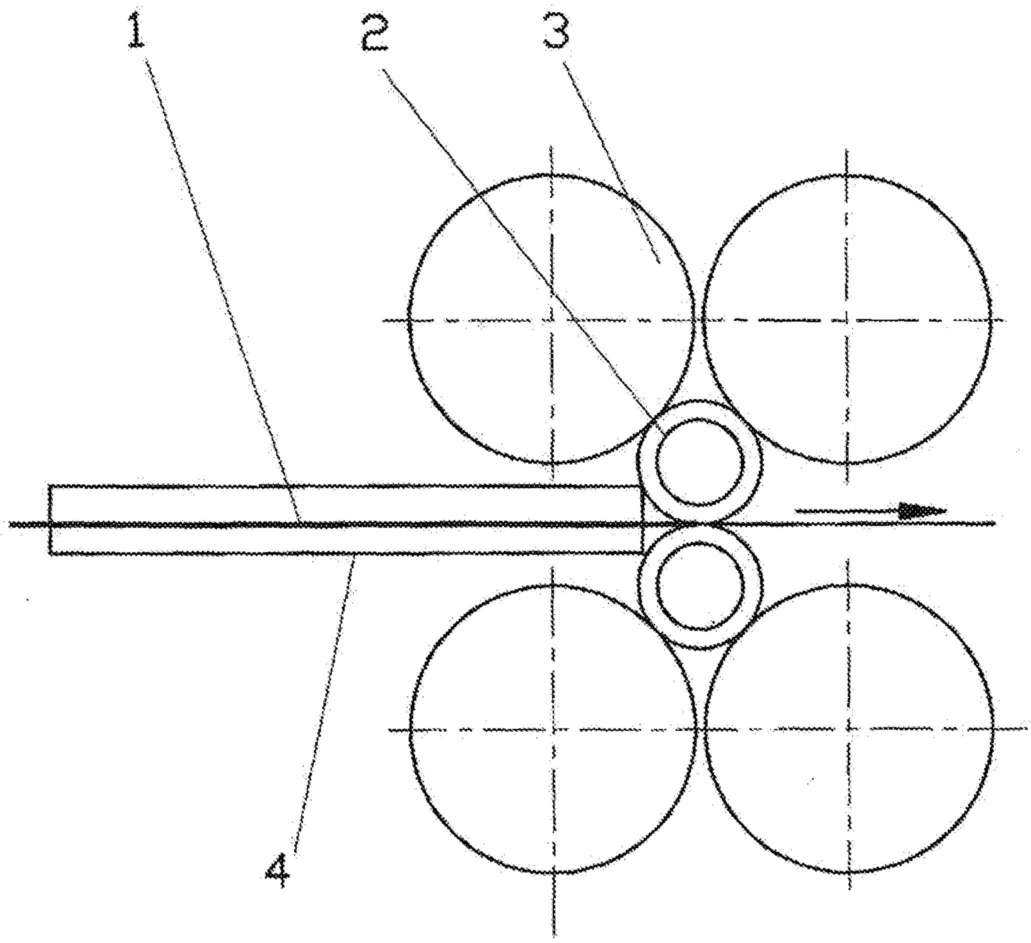
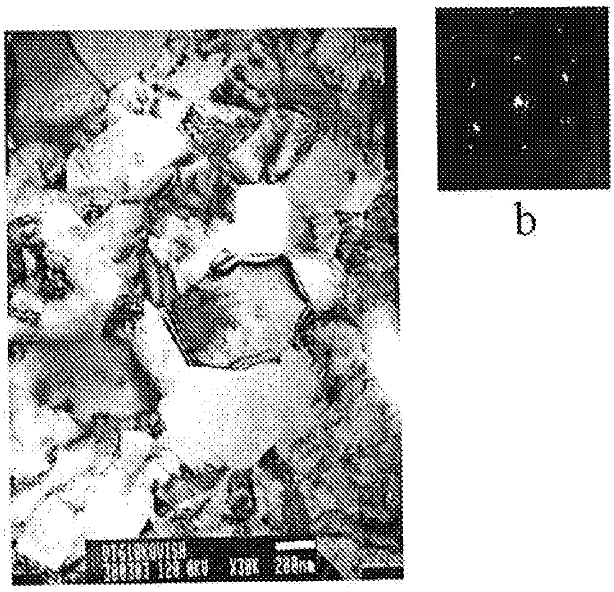


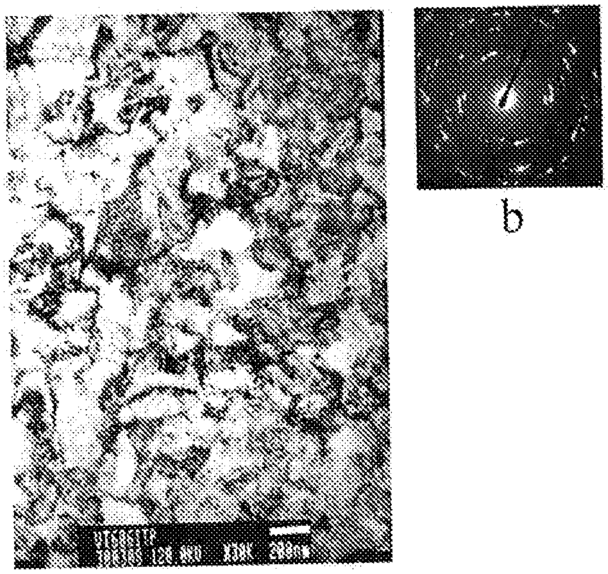
Fig. 1



b

a

Fig. 2



b

a

Fig. 3

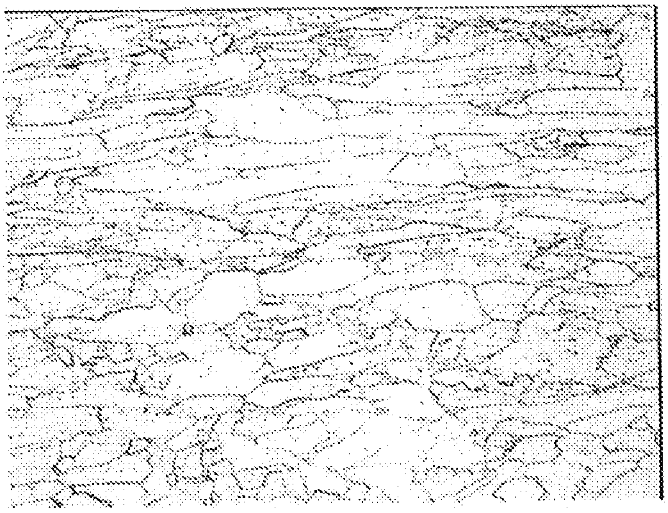


Fig. 4

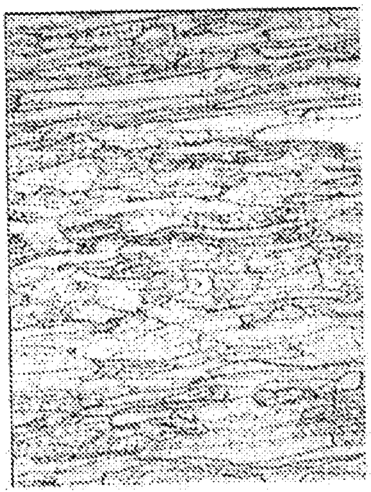


Fig. 5

Fig. 6

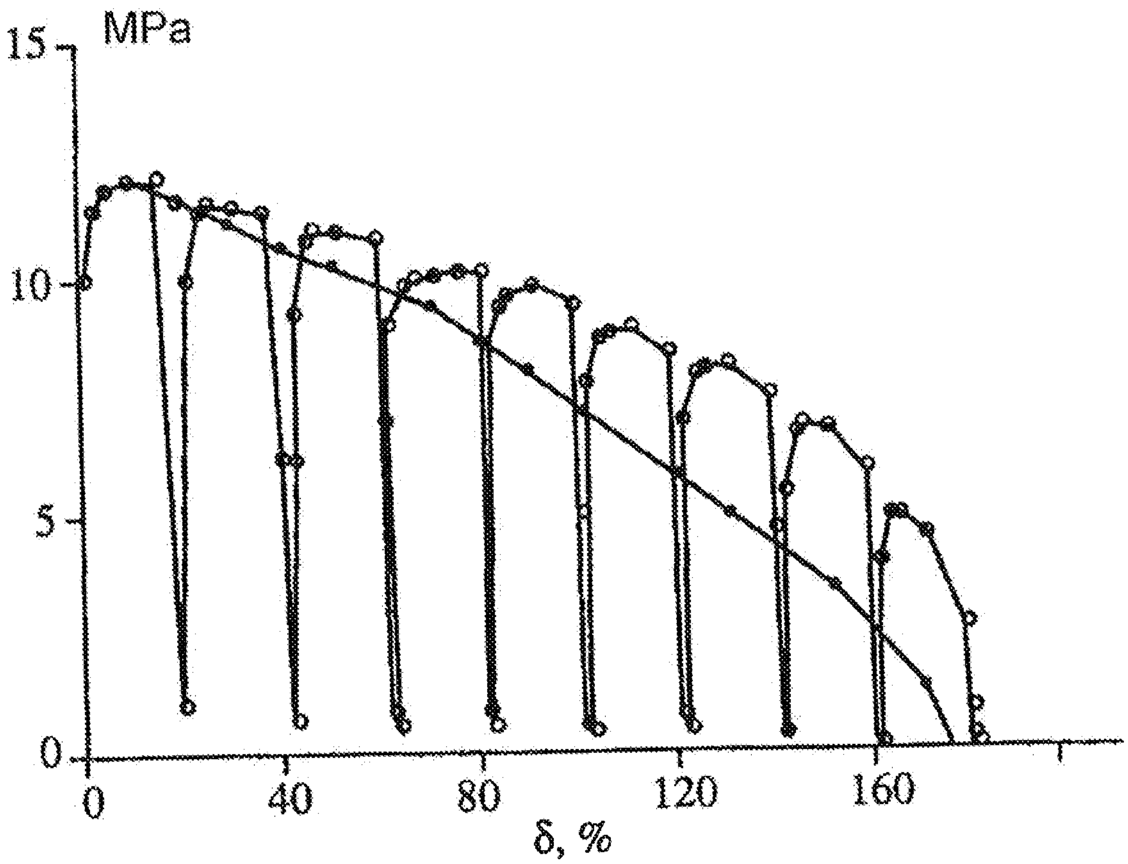
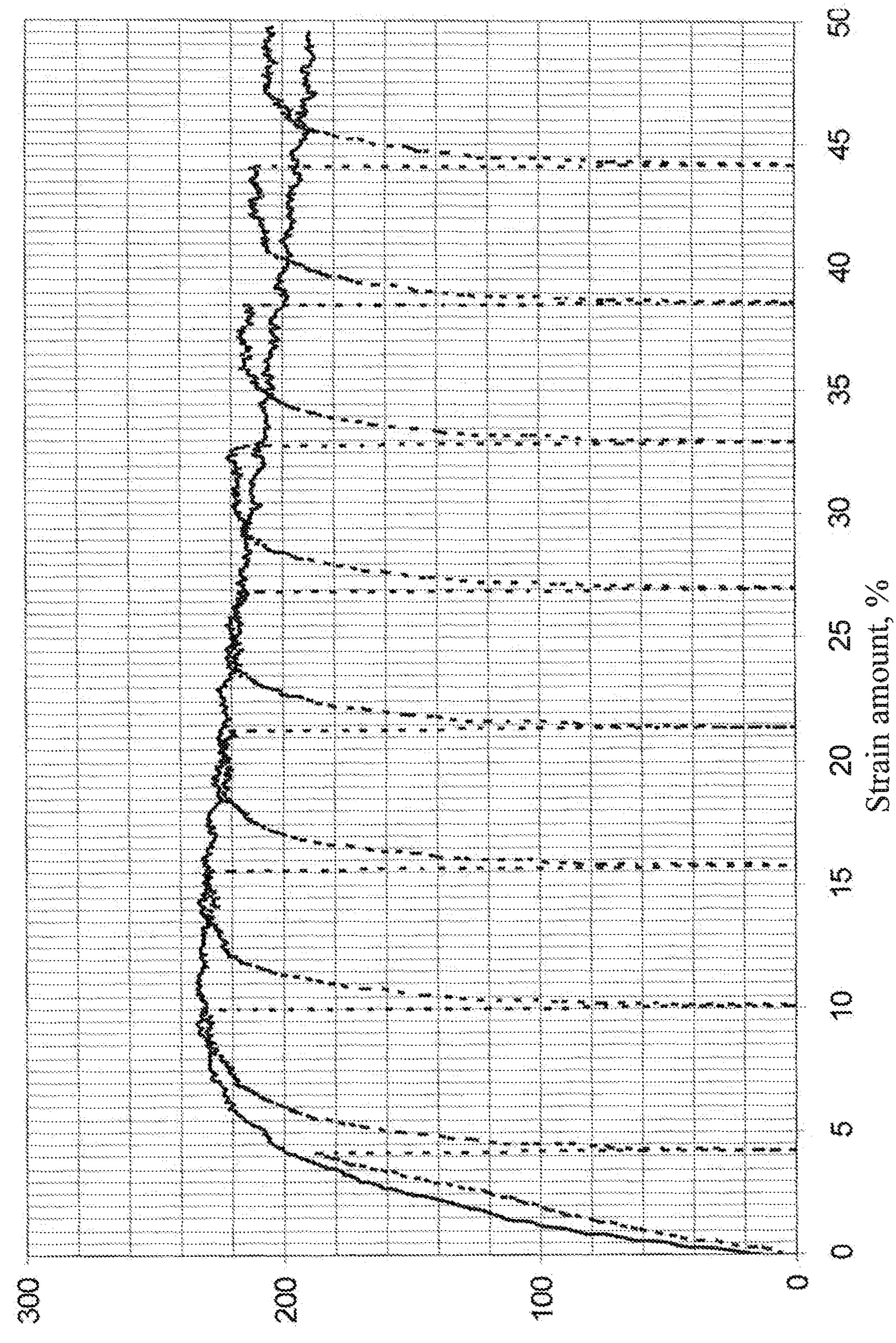


Fig. 7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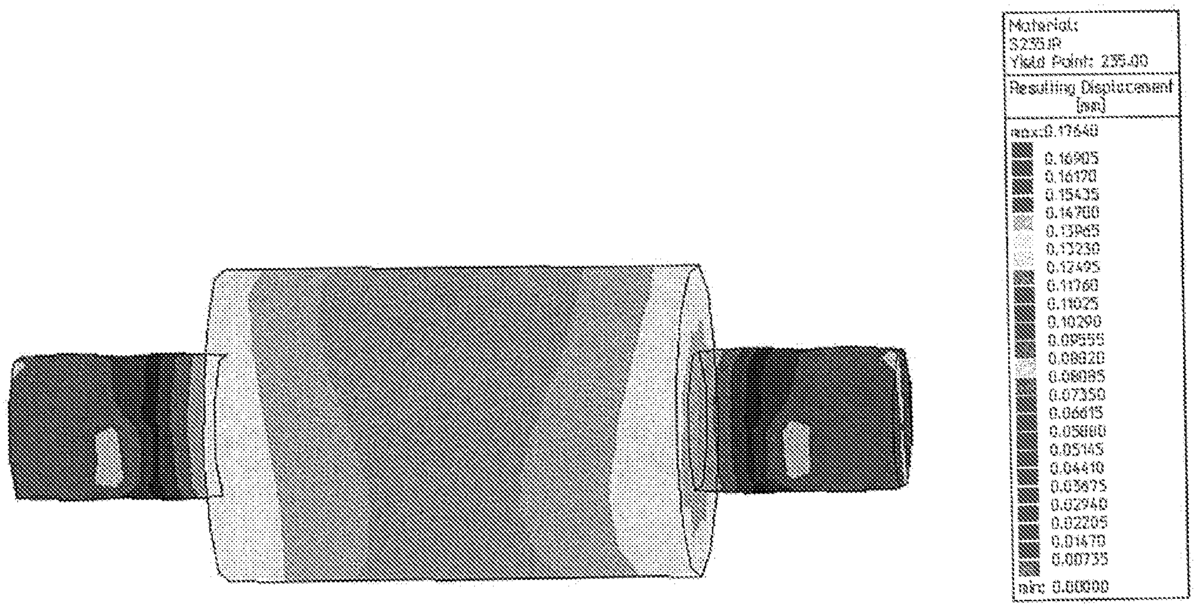
200
100
0
Strain amount, %
0
5
10
15
20
25
30
35
40
45
50

Fig. 8


Material:
S235JR
Yield Point: 235.00
Resulting Displacement
[mm]
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0.16905
0.16170
0.15435
0.14700
0.13965
0.13230
0.12495
0.11760
0.11025
0.10290
0.09555
0.08820
0.08085
0.07350
0.06615
0.05880
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0.02940
0.02205
0.01470
0.00735
min: 0.00000

Fig. 9

INTERNATIONAL SEARCH REPORT

International application No.
PCT/RU2007/000123

A. CLASSIFICATION OF SUBJECT MATTER

C22F 1/18 *(2006.01)*
B21B 3/00 *(2006.01)*

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C22C 1/00 1/10, 1/18, B21B 3/00

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

RUPAT, RUPAT, OLD, PAJ, Esp@cenet

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	RU 2224047 C1 (INSTITUT PROBLEM SVERKHPLASTICHNOSTI METALLOV RAN) 20.02.2004	1-12
A	RU 2058418 C1(INSTITUT PROBLEM SVERKHPLASTICHNOSTI METALLOV RAN) 20.04.1996	1-12
A	RU 2250806 C1 (OAO VERKHNESALDINSKOE METALLURGICHESKOE PROIZVODSTVENNOE OBIEDINENIE (VSMPO)) 27.04.2005	1-12
A	JP 63230858 A (SUMITOMO METAL IND) 27.09.1988	1-12

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

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"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
23 May 2007 (23.05.07)	31 May 2007 (31.05.07)
Name and mailing address of the ISA/ RU	Authorized officer
Facsimile No.	Telephone No.

Form PCT/ISA/210 (second sheet) (July 1998)

REFERENCES CITED IN THE DESCRIPTION

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