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(54) METHOD FOR THE MANUFACTURING OF EXTRUDED PROFILES THAT CAN BE ANODIZED WITH HIGH GLOSS SURFACES, THE PROFILES BEING EXTRUDED OF AN AGE HARDENABLE ALUMINIUM A 7XXX ALLOY THAT CAN BE RECRYSTALLIZED AFTER COLD DEFORMATION

VERFAHREN ZUR HERSTELLUNG VON EXTRUDIERTEN PROFILEN, DIE MIT HOCHGLÄNZENDEN OBERFLÄCHEN ANODISIERT WERDEN KÖNNEN, WÄHREND DIE PROFILE AUS AUSHÄRTBARER, NACH KALTUMFORMUNG REKRISTALLISIERBARER 7XXX-ALUMINIUMLEGIERUNG EXTRUDIERT WERDEN

PROCÉDÉ DE FABRICATION DE PROFILÉS EXTRUDÉS POUVANT ÊTRE ANODISÉS COMPORTANT DES SURFACES À HAUT BRILLANT, LESDITS PROFILÉS ÉTANT EXTRUDÉS À PARTIR D'UN ALLIAGE 7XXX D'ALUMINIUM DURCISSABLE PAR VIEILLISSEMENT POUVANT ÊTRE RECRISTALLISÉ APRÈS DÉFORMATION À FROID

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Description

[0001] The present invention relates to a method for the manufacturing of extruded profiles that can be anodized with high gloss surfaces, the profiles being extruded of an age hardenable aluminium alloy that can be recrystallized after cold deformation, for example 7xxx (Al-Mg-Zn) alloys. The oxide layer (Al203) formed during anodizing is build up by dissolving the outer layer of the aluminium. For each 3 μιτι of oxide layer formed 2 um of the aluminium is dissolved. Since the oxide layer is bulkier than the aluminium the total thickness will then increase by 1 um. In order to obtain high gloss of an anodized aluminium product it is important to keep the amount of constituent particles with a diameter larger than approximately 0.3 um (S. Wernick, R. Pinner and P.G. Sheasby, The Surface Treatment and Finishing of Aluminium and its Alloys, ASM INTERNATIONAL, FINISHING PUBLICATIONS LTD, Fifth Edition Vol 1, 1987, p. 143) at a low level, since these particles will be embedded in the anodized layer and cause a reduction in the gloss. The most important factor to achieve this is to keep the amount of Fe at a low level, since primary AlFeSi particles are insoluble in the aluminium matrix. Typically, alloys used for high gloss products have a maximum limit of Fe around 0.12 wt%. Gloss is thus also reduced with increasing thickness of the oxide layer formed during anodizing since more particles then will be embedded. Moreover the process parameters used during anodizing also affect the gloss.

[0002] EP3129517 relates to a method for manufacturing products with anodized high gloss surfaces from extruded profiles of Ai-Mg-Si or AS-Mg-Si-Cu. The patent is silent on the use of 7XXX alloys.

[0003] In EP0507411 AIMgSi alloys of the 5XXX series are rolled to sheets with a smooth surface after recrystallisation anneal where the resulting grain size is <50 um. No process for producing profiles is thought of. The content of Zn in the alloys is below 0.25%.

[0004] US 2008/173378 A1 relates to a method of manufacturing a wrought aluminum alloy product of an AA7000-series alloy comprising 3 to 10% Zn, 1 to 3% Mg, at most 2.5% Cu, Fe <0.25%, and Si ≤0.12%, the method including hot working the stock by one or more methods selected from the group consisting of rolling, extrusion, and forging.

[0005] Hardening precipitates are formed during the artificial ageing process (e.g. β"-MgSi) from the addition of Mg and Si. If Cu is added in sufficient amount other phases than β" may form (e.g. Q' and L) {Calin D. Marioara, et. al., Improving Thermal Stability in Cu-Containing Al-Mg-Si Alloys by Precipitate Optimization, METALLURGICAL AND MATERIALS TRANSACTIONS A, March 2014). These hardening precipitates are much smaller than 0.3 um and are therefore not reducing the gloss in the same way as the primary AlFeSi particles. The strength requirement for the alloy determines the necessary amount of Mg, Si and Cu in the alloy. In order to maximize the gloss it is necessary to process the material in a way where precipitation of larger non-hardening phases (e.g. β'-MgSi and p-Mg2Si) of Mg, Si and Cu is avoided. This is easiest to obtain for 6060 and 6063 type of alloys where the Mg and Si contents are relatively low. Higher alloyed material requires higher temperatures in the extrusion or solutionising processes and faster cooling afterwards to avoid precipitation of such particles.

[0006] Alloying elements such as Mn, Cr, Zr or Sc can be added to form dispersoid particles during homogenization. Frequently, these elements are added in high amounts in order to prevent recrystallization in the extruded profile. However, it can be beneficial to add these elements in smaller amounts to only have some dispersoid particles in the alloy in order to avoid grain growth during homogenization and after the recrystallization process occurring in the extrusion process or in a separate recrystallization and solutionising process for the cold deformed material. The size of these particles is typically between 0.01-0.2 μηι. Thus, such particles can be added, at least in a relative low number, without significantly affecting the gloss. However, the number of dispersoid particles should not be so high that the exposed areas of the profile surface get a mixture of a non- recrystallized and a recrystallized structure or a fully recrystallized structure with a large and uneven grain size. Addition of elements that form dispersoid particles can also give an unwanted color of the anodizing layer, or they can give an unwanted surface appearance due to a strong texture of the recrystallized grains.

[0007] If an anodized surface contains large grains the individual grains can be detected by the naked eye. This surface defect is frequently called mottling. The best surface appearance is obtained when the average grain size is smaller than approximately 70 um and the grains mainly are randomly orientated.

[0008] If the processing of the material is satisfactory there will be no large β'-MgSi or -Mg2Si particles present in the extruded and aged profile samples. In such a case the gloss will be more or less proportional to the amount of Fe in the alloy for a given anodizing process. To maximize the gloss one would like to minimize the Fe content. Reducing the Fe content will increase the price of the aluminium since it will be more costly to produce. It will require alumina with low Fe and low contribution of Fe from the anodes. The processing in the electrolysis and the casthouse also has to be adapted in order to produce aluminium with very low Fe content. The main problem by using very low Fe contents is, however, the ability to control the grain size in the billet and in the extruded profile.

[0009] From Japanese patent publication No. 10-306336 is known an aluminium alloy extruded material having high surface gloss after anodic oxidation treatment where the surface gloss allegedly is made uniform by specifying the number of the particles of Mg2Si participated in the matrix. This is obtained with a specific heat treatment procedure prior to and after extrusion.

With the present invention it is provided a method for the manufacturing of products that can be anodized high gloss surfaces from extruded profiles of for example 7xxx alloys, with excellent mechanical properties and at low costs.

[0010] The method according to the invention is characterized by the features as defined in the accompanying independent claim 1. Further embodiments are defined in the subordinate claims 2 - 14.

[0011] The invention will be further described in the following by way of examples and with reference to the drawings and figures where:

Fig. 1 is a photo of a quarter of a macro etched billet slice (0228 mm in diameter) with abnormal grains,

Fig. 2 light optical micrograph showing a typical grain structure of a 6060 alloy through the thickness of a thick solid shape extruded profile. The sample is taken from a transverse cross section and is anodized and viewed in polarized light,

Fig. 3 is a principal sketch of an industrial processing line for performing the cold rolling and the annealing process described in the present invention,

Fig. 4 shows light optical micrographs of samples from example 1 showing the grain structure of a 6060 alloy in the middle of the transverse cross section for the as extruded profile and for the samples that were cold rolled to give 10, 20, 40 and 60% reduction in the thickness prior to annealing. All samples are anodized and viewed in polarized light,

Fig. 5 shows grain structure in an as cast billet (o95 mm diameter) without grain refiner, which was used in example 2 of the present application. Picture of a macro etched billet slice to the left and anodized sample viewed in polarized light in a light optical microscope to the right,

Fig. 6 are light optical micrographs showing the AlFeSi particles in a homogenized billet cast without grain refiner (upper picture) and in a homogenized billet cast with grain refiner (lower picture). The position of the samples in the billet is approximately half radius, is a light optical micrograph of an as extruded sample in example 2 of the application, showing the grain structure in a transverse cross section close to the surface. Anodized and viewed in polarized light,

Fig. 8 shows light optical micrographs of samples from example 2, showing the grain structure in the middle of the transverse cross section for the as extruded profile and the samples that were cold rolled to give 20, 30, 40 and 50% reduction in the thickness prior to annealing. All samples are anodized and viewed in polarized light,

Fig. 9 shows further light optical micrographs of samples from example 2 of the present application, showing the grain structure in the middle of the cross section for samples that were cold rolled to 40% reduction in the thickness prior to annealing in air (upper) and in a salt bath (lower). Both samples are anodized and viewed in polarized light, Fig. 10 light optical micrograph of a sample of alloy 7030 from example 3, showing the grain structure through the transverse cross section of a profile that has been cold rolled to 10% reduction in thickness and subsequently flash annealed in a salt bath. The sample is anodized and viewed in polarized light,

Fig. 11 light optical micrographs of samples of alloy 7030 from example 3, showing the grain structure in the middle of the transverse cross section for the as extruded profile and the samples that were cold rolled to give 20, 30, 40 and 50% reduction in the thickness prior to annealing. All samples are anodized and viewed in polarized light,

Fig. 12 the grain structure towards one end of the transverse profile cross section of a 40% cold rolled and annealed sample of alloy 7030. The sample is anodized and viewed in polarized light.

[0012] When the Fe content is below approximately 0.10 wt% in a 6060 or 6063 type of alloy the chance of getting abnormal grains (grains that grow and consume other grains that were formed during casting) in the billet during homogenization becomes very high. Therefore, a grain size of several centimeters is very common in billets of alloys with very low amounts of Fe. An example of abnormal grains in a homogenized billet with low Fe content is shown in Figure 1.

[0013] For a 7xxx alloy the homogenizing temperatures are normally lower than for a 6060 alloy. This may reduce the problem with abnormal grain growth during homogenization.

[0014] The billet grain size will probably not affect the grain size in the extruded profile much if the extent of deformation is high, for example when extruding thin walled hollow profiles. For solid shapes, and especially for thick walled profiles, the billet grain size will most likely affect the grain size in the extruded profile. An additional challenge is that the billet temperature needs to be rather high in order to dissolve the Mg2Si particles, and a high billet temperature makes it more difficult to obtain a small grain size after extrusion.

[0015] In an extruded profile, one usually sees a surface layer of mainly randomly oriented grains and typically one or a few grains in thickness. Underneath this layer one typically finds a region of larger grains. The thickness of this layer varies, and is usually thicker for a thick walled and wide solid shape profile and thicker towards the back end of the extruded length. An example of a typical grain structure in a cross section of a thick walled industrially extruded profile can be seen in Figure 2. Below the layer of larger grains the grain structure is typically more homogeneous. The grains in the homogeneous center region of the cross section are predominantly aligned in one direction, with a strong cube texture. This is often seen in a micrograph of the grain structure in the cross section by small differences in the color of the grains. More and more consumer electronics like mobile phones, tablets and lap tops are made of aluminium from extruded profiles. If the profile surface could have been used without any machining the grain structure in the anodized surface would probably be okay in most cases. However, very often there is a need to machine the extruded profile to make the shape and the dimensional tolerances of the final product. In that case the exposed surface can consist of grains from the coarse grain layer beneath the surface layer of the extruded profile. Due to this the entire coarse grain layer has to be removed before starting to machine the shape of the final product. The thickness of the layer that has to be removed due to coarse grains will vary with the width of the profile and the extrusion conditions and is typically in the range of 0.2 to 1 mm.

[0016] The present invention deals with the task to get a homogeneous grain structure with an average grain size below approximately 70 um irrespective of the Fe content, the grain size in the billet prior to extrusion and the extrusion conditions. Solid shape profiles which are blanks for consumer electronics will be more or less flat, but could possibly have some features in the cross section in order to save material and machining. Such profiles are therefore very well suited for cold rolling after extrusion. By cold rolling a profile by a minimum of 10% followed by flash annealing a new recrystallization process will take place. With sufficient deformation and a proper annealing process the resulting grain structure will be homogeneous over the cross section with a much more random orientation of the grains than in the as extruded profile. The grain size will in addition to the alloy content, depend on the degree of cold deformation, the annealing temperature, the heat up conditions and the time at the annealing temperature. In an alloy with very low Fe and no dispersoid particles the recrystallization will take place at a low temperature, most likely during heating to the annealing temperature. One issue will then be to avoid grain growth at the annealing temperature when there are almost no particles in the material to pin the grains.

[0017] The annealing temperature should preferably be above the solvus temperature for Zn2Mg particles (7xxx) in order to avoid particles that can reduce the strength and the gloss of the anodized material. In addition, the time at this annealing temperature should be as short as possible in order to avoid grain growth. Therefore, the material should be processed through extrusion in a way that Mg2Si or Zn2Mg particles are avoided. This means sufficiently high billet temperature in combination with a high enough exit temperature from extrusion and fast cooling of the profile after extrusion. With no Mg2Si or Zn2Mg particles in the material prior to cold rolling and annealing there is no need for a holding time for the material at the annealing temperature.

[0018] The consequence of annealing at temperatures below the solvus temperature will be that Mg-Si containing precipitates or Zn2Mg precipitates larger than approximately 0.3 um may form. These particles will contribute to a reduction in the gloss and in the strength of the material. The amount of this reduction will depend on the actual time-temperature history during the flash annealing and cooling operation and the composition of the alloy. An industrial process to perform the cold rolling and the annealing process could be done as shown schematically in Figure 3. The cold rolling station should be followed by a station for performing fast heating to the annealing temperature. Using induction heating is probably the best way to do this. With enough power and induction coils that fit the shape of the profile and good process control, it should be possible to heat the material to a temperature around 500°C (depending on the composition and thereby the solvus temperature of the alloy) within a very short time and with sufficient accuracy in temperature.

[0019] In order to avoid precipitation of Mg-Si containing precipitates or Zn2Mg precipitates larger than approximately 0.3 um the profile needs to be cooled rather rapidly down to room temperature. The reason for this is described in a previous section. Thus, preferably according to the present invention, the profile is flash annealed with a heating time of maximum two minutes to a temperature of between 450 - 530 °C and held at this temperature for not more than 5 minutes and subsequently quenched.

[0020] After the annealing operation there is probably a need to remove residual stresses from the quenching operation. The best way to this would probably be to stretch the material in way similar to what is done after extrusion. After the annealing process the final ageing of the material can for example be done with the patented dual rate ageing cycle (U. Tundal and O. Reiso, EP 1 155 161 B1) to get maximum strength with minimum amount of alloying elements. The invention will be further described in the following by way of examples.

Comparative example 1

[0021] Billets with diameter 95 mm were cast in a lab casting facility using the Hycast hot-top gas-slip technology (as described in EP 0 778 097 B1) and a T1B2 based grain refiner. The composition of the alloy is shown in Table 1.

[0022] Table 1. Chemical composition of the alloy used in example 1 Image available on "Original document"

[0023] The billets were homogenized at 575°C for 2 hours and 15 minutes followed by cooling at a rate of approximately 400°C per hour. Extrusion of the billets was performed at an 8 MN laboratory extrusion press with a 100 mm diameter container to a profile with 5x40 mm<2>cross section. The billet preheating temperature was approximately 500°C and the extrusion speed 20 m/min. After extrusion the profile was quenched in water. A 50 cm long piece from the front part of the extruded profile was cold rolled to give 10, 20, 40 and 60% reduction in the thickness. The samples that were cold rolled to different thicknesses were then annealed in a salt bath which had been preheated to 500°C. A hole was drilled into each of the samples to fit a thermocouple. The heating time to temperature was in the range 5 - 10 seconds, depending on the thickness of the sample. When a sample was put into the salt bath a holding time of 10 seconds started when the temperature reached 490°C. After annealing the samples were quenched in water. The cross section of all samples (in all examples) were prepared by grinding and mechanical polishing with a final step using 1 um diamond paste. In order to make the grains visible in polarised light, anodising was performed in a Struers Lectropol-5 with the following parameters. Voltage: 45 V; Flow rate: 3; Temperature: -5°C; Time: 2 minutes. The electrolyte had the following ingredients: 74% distilled water; 24% ethanol; 1 % HBF4(35%); 1 % HF (40%).

[0024] Prior to extrusion the billets had an even and small grain size. The as extruded sample in Figure 4 shows a homogeneous grain size throughout the cross section. In this case there is no significant coarse grain layer below the surface. This is maybe because the sample is smaller than the sample shown in Figure 2 and maybe also because it is taken from the front part of the extruded length. It is evident that the grains under the randomly oriented layer of grains in the profile surface area are predominantly aligned in one direction since the color contrast between the grains is low.

[0025] As can be seen from the large color contrast, the cold rolled and annealed samples show a much more random orientation of the grains than the as extruded sample. This confirms that these samples are fully recrystallized after annealing. The samples that were cold rolled to 10 and 20% reduction in thicknesses clearly have an uneven grain structure with the largest grains in the middle of the cross section. The samples that were cold rolled to 40 and 60% reduction in thicknesses have an even grain structure throughout the cross section. The grain sizes of the samples shown in Figure 4 (measured 250 um below the surface of the cross sections) are shown in Table 2. Table 2. Average grain sizes of the 6060 alloy samples in example 1 as measured 250 um below the surface of the cross section. The as extruded grain size is very uncertain due to the very low contrast between the individual grains.

[0026] 10% cold rolled 20% cold rolled 40% cold rolled 60% cold rolled

As extruded

+ annealed + annealed + annealed + annealed

-87 um 79 um 60 um 44 um 33 um

<u>Comparative</u> Example 2

[0027] Billets with diameter 95 mm were cast in a lab casting facility using the Hycast hot-top gas-slip technology without using a grain refiner. A picture of a macro etched billet slice is shown in Figure 5 together with a micrograph showing an anodized sample viewed in polarized light in the light optical microscope. Towards the surface there are some relatively large equiaxed grains, but a large part of the cross section of the billet slice consists of feather crystals. The composition of the alloy is shown in Table 3.

[0028] Table 3. Chemical composition of the alloy used in example 2 Image available on "Original document"

[0029] The cast billets were homogenized at 575°C for 2 hours and 15 minutes followed by cooling at a rate of approximately 400°C per hour. Micrographs of the particle structure in the billets from the two different alloys in examples 1 and 2 are shown in Figure 6. The material cast without grain refiner (upper picture) shows Fe containing particles (mainly a-AlFeSi) that are smaller and much more evenly distributed than the Fe containing particles (mainly β-AlFeSi) in material cast with grain refiner (lower picture). In the latter case the AlFeSi particles mainly are located at the grain boundaries. In both cases the Fe/Si ratio is very low, which makes β-AlFeSi particles very stable in the homogenizing process. A particle structure as shown in the material cast without a grain refiner would be beneficial in avoiding alignment of particles and possible visible dark lines in the extruded and anodized high gloss surface. The billets were extruded at an 8 MN laboratory extrusion press with a 100 mm diameter container to a profile with a cross section of 5x40 mm<2>. The billet preheating temperature was approximately 500°C and the extrusion speed 20 m/min. After extrusion the profile was quenched in water.

[0030] A 100 cm long piece from the back part of the extruded profile was cold rolled to give 20, 30, 40 and 50% reduction in the thickness. The samples that were cold rolled to different thicknesses were then annealed in a salt bath which had been preheated to 500°C. A hole was drilled into each of the samples to fit a thermocouple. When a sample was put into the salt bath the holding time of 10 seconds started when the temperature reached 490°C. After annealing the samples were quenched in water. In addition one sample of the material cold rolled to 40 % reduction in thickness was held 5 minutes at 500°C. Yet another sample of the material cold rolled to 40% reduction in thickness was heated in an air circulating oven at a considerably lower heating rate to the annealing temperature than that obtained in a salt bath. A micrograph of the as extruded sample is shown in Figure 7. It seems like some of the grains below the surface are considerably larger than 100 um, which could give some unwanted effects in the surface appearance. Inside the surface region the grains are strongly aligned in one direction, which gives very little contrast between each individual grain in the micrograph.

[0031] Figure 8 shows micrographs of the grain structure in the as extruded sample as well as samples that have been cold rolled 20, 30, 40 and 50% and thereafter annealed. As also seen in example 1, one can see from the large color contrast that the cold rolled and annealed samples show a much more random orientation of the grains than the as extruded sample. The sample that was cold rolled to 20% reduction in thickness clearly has an uneven grain structure with the largest grains in the middle of the cross section. The sample cold rolled to 30% reduction in thickness has smaller grains and a more even grain structure, but the grains in the middle still are somewhat larger than those towards the surfaces. The samples that were cold rolled to 40 and 50% reduction in thicknesses have a smaller grain size and an even grain structure throughout the cross section. As also shown in Table 4 (below) the grain size seems to be similar for the samples cold rolled to 40 and 50% reduction in thicknesses.

[0032] Table 4 (below). Average grain sizes of the 6060 samples in example 2 as measured 250 μητι below the surface of the cross section. The as extruded grain size is very uncertain due to the very low contrast between the individual grains. Image available on "Original document"

[0033] The sample that was cold rolled to 40% reduction in thickness and held at 500°C for 5 minutes did not show any grain growth. The reason for this is probably that the number of AlFeSi-particles is high enough to prevent grain growth. With even lower Fe contents than 0.09 wt% a holding time of 5 minutes at this temperature could cause grain growth in the sample.

[0034] Figure 9 shows that the sample heated in an air-circulating furnace (6-7 minutes heating time) has a more uneven grain structure and a slightly larger grain size than the sample that was rapidly heated (5-10 seconds) in a salt bath up to the solutionizing temperature. The reason for this is probably linked to precipitation of Mg-Si particles at the grain boundaries, which are pinning the nuclei for new grains during the heat up process. In a sample which is slowly heated in air there is enough time for precipitation of Mg-Si particles to prevent the nuclei for new grains from growing until the particles start to dissolve again, i.e. when the sample is approaching the solvus temperature of the alloy. In this process some grains will probably start to grow earlier than others and therefore get larger, resulting in an uneven grain structure when the recrystallization process is complete. Example 2 shows that it is beneficial to heat the cold rolled sample fast to the solutionizing temperature to obtain an even grain size and that a holding time of only 10 seconds is sufficient to obtain a fully recrystallized grain structure. Example 2 also shows that the final grain structure in the blanks could be perfect for providing attractive high gloss anodized surfaces even though the billet grain structure is regarded as being far from optimum when it is cast without grain refiner.

Example 3 (according to the present invention)

[0035] Billets with diameter 95 mm of a 7030 alloy were cast in a lab casting facility using the Hycast hot-top gas-slip technology and a T1B2 based grain refiner. The chemical composition of the alloy is shown in Table 5.

[0036] Table 5. Chemical composition of the alloy used in example 3 Image available on "Original document"

[0037] The billets of the 7030 alloy were homogenised for 4 hours at 500°C. The billets were extruded at an 8 MN laboratory extrusion press with a 100 mm diameter container to a profile with a cross section of 5x40 mm<2>. The billet preheating temperature was approximately 500°C and the extrusion speed 12.5 m/min. After extrusion the profile was quenched in water.

[0038] A 100 cm long piece from the extruded profile was cold rolled to give 20, 30, 40 and 50% reduction in the thickness.

[0039] The cold rolled samples at different thicknesses were then, one by one, put into a salt bath that had been preheated to 500°C. With a thermocouple drilled into each sample it was possible to monitor the temperature of the sample. All samples were held approximately 10 seconds at a temperature above 495°C before quenching in water. The heating rates of the samples depended on the thickness, but in all cases the heating time was less than 10 seconds.

[0040] Figure 10 shows the grain structure through the transverse cross section of a 7030 sample that has been cold rolled to a 10% reduction in thickness and subsequently flash annealed in a salt bath. As can be seen, the grain structure is very uneven, with some grains being more than 500 μιτι in diameter. This shows that 10% deformation by rolling is too little to create a uniform grain structure through the cross section of the material.

[0041] Figure 11 shows the grain structures through the thickness of a transverse cross section of an as extruded 7030 profile as well as of samples that have been cold rolled to 20, 30, 40 and 50% reduction in thicknesses and subsequently flash annealed. The grain structure of the as extruded sample is significantly coarser than the grain structure in the 6060 alloy. This could either be a result of the lower extrusion speed used for the 7030 alloy or a higher solute drag from the high amount of Mg, Zn and Cu in this alloy. The sample rolled 20% show a slightly coarser grain structure than the as extruded sample, especially in the middle of the cross section. The sample rolled 30% has a grain structure that would fulfil the requirements of a grain size below about 70μηι, but the grain size in the middle is somewhat larger than towards the surface. The grain structures in the samples cold rolled by 40 and 50% show a very nice grain structure throughout the cross section. Based on the visual appearance of the anodized grain structures, the as extruded sample of the 7030 alloy does not seem to have the same strong cube texture as the 6060 alloy.

[0042] As shown in Figure 12, the grain structure is also very uniform towards the ends of the cross section when a sample of a 7030 alloy has been cold rolled by 40% before the flash annealing process. The grain sizes of some of the samples depend on the depth below the surface of the cross section. Some samples have very coarse grains in the middle of the cross section and finer grains towards both surfaces. A typical machining depth to remove the coarse surface grain layer in a small profile like this would be around 250 μητι, and this depth was chosen for the grain size measurements. In Table 6 (below) the grain sizes for the alloy of Example 3 are listed. By looking at these grain size measurements alone, all the samples seem to fulfil the requirement of a grain size below approximately 70 um. However, the pictures in Figure 1 1 give a better overview of the grain structures in the samples. From the grain size measurements and the pictures one can state that all samples with 30, 40 and 50% cold rolling followed by annealing fulfil the grain structure requirements for the alloy. With less deformation the grain structure seems to be too uneven.

[0043] Table 6. Average grain sizes of the 7030 alloy samples in example 3 as measured 250 μητι below the surface of the transverse cross section. Image available on "Original document"

[0044] The main benefit of the present invention is that it is possible to obtain a grain structure with an even grain size and a close to random texture throughout the cross section of a profile irrespective of the grain size in the profile after it has been extruded and thus also irrespective of the grain structure of the billet before extrusion. This improvement in grain structure is obtained by cold rolling deformation of the extruded profile followed by flash annealing.

[0045] An extruded thick walled flat profile will in most cases have a coarse grain layer that according to the state of the art has to be removed in order to obtain a smooth anodized surface with a minimum of defects in the final product. The amount of material that would have to be removed in the as extruded cross section is typically in the range 7-15%.

[0046] Moreover, the cold rolling will ensure a very accurate thickness and flatness of the profile, and for that reason considerably reduce the need for machining. An extruded profile will have much more variation in the thickness, typically ± 0.15 mm (the variation could also be higher for very wide profiles, especially for 7xxx alloys).

[0047] Since the grain size in the billet and the extruded profile is of little importance for the resulting grain size in the cold rolled and annealed blanks there is a possibility of casting the billets with a minimum or even completely without the use of a grain refiner. In order to avoid center cracks in the billets in the startup of the cast it could be beneficial to add some grain refiner in the first metal to cast. The grain refiner itself could be a source for inclusions that can cause failures in the anodized surface. Another benefit of not using a grain refiner is that the melt cleaning with the use of ceramic foam filters will be more effective on other type of inclusions (Nicholas Towsey, Wolfgang Schneider and Hans-Peter Krug, A comprehensive study of ceramic foam filtration, 7th Australasian Asian Pacific Course & Conference, Aluminium Cast House Technology: Theory & Practice, P. Whiteley and J. Grandfield (TMS: 2001)

[0048] The possibility of reducing the Fe content and still obtain an adequate grain structure will significantly improve with the use of the present invention. The lower Fe content can either be used to improve the gloss, or to keep the current gloss but add a thicker and more wear resistant oxide layer to the anodized product. The latter will make the product more durable.

[0049] Even though there is extra cost associated with the cold rolling and annealing process to obtain the uniform and random grain structure, this will probably be more than compensated for by the savings due to reduced machining and reduced material consumption.

Claims

1. Method for the manufacturing of extruded profiles that can be anodized with high gloss surfaces, the profiles being extruded of an age hardenable aluminium alloy that can be recrystallized after cold deformation, where the alloy initially is cast to extrusion billets, where the billets are homogenized at a holding temperature between 480°C and 620°C and soaked at this temperature for 0-12 hours, where after the billets are subjected to cooling from the homogenization temperature at a rate of 150°C/h or faster,

a) the billets are preheated to a temperature between 400 and 540°C and extruded and cooled rapidly down to room temperature ,
b) deforming the profile more than 10% by a cold rolling operation, where after c) the profile is flash annealed with a heating time of maximum two minutes to a temperature of between 400 - 530 °C and held at this temperature for not more than 5 minutes to obtain an average grain size of about 100 μm or less, and subsequently quenched, and
d) the profile is finally aged, wherein the alloy is a 7xxx alloy containing in wt.%:

Si: 0,00-0,30
Mg: 0,50 -2,00
Fe: 0,00-0,15
Cu: 0,00 -0,30
Mn: 0,00 - 0,20
Cr: 0,00-0,10
Zr: 0,00 - 0,20
Sc: 0,00-0,10
Zn: 3,00 - 7,00

Ti: 0,00 - 0,05, and
including incidental impurities and balance Al.

2. Method according to claim 1, **characterised in that** the profile between step c) and d) is further subjected to a cold deforming operation, preferably by stretching to remove residual stresses from cooling.

3. Method according to claim 1, **characterised in that** the profile between step c) and d) is cut into blanks that is cold formed to a shape that saves material and machining time to produce the final product.

4. Method according to claim 1, **characterised in that** the profile according to step b) is deformed more than 20%.

5. Method according to claim 1, **characterised in that** the profile according to step b) preferably is deformed between 30 and 50%.

6. Method according to claim 1 , **characterised in that** the profile is flash annealed according to step c) with a heating time of maximum 20 seconds to a temperature between 400 - 530 °C and held at this temperature for not more than 1 minute.

7. Method according to claim 1, **characterised in that** the flash anneal heating according to step c) is obtained by induction heating of the profile.

8. Method according to claim 1 , **characterised in that** the flash anneal heating according to step c) is obtained by subjecting the profile to a salt bath or other convection or radiation heating means providing high heating rates.

9. Method according to claim 1, **characterised in that** the alloy is cast without the use of grain refiner, except in the start-up of the casting operation.

10. Method according to claim 1, **characterised in that** the ageing, step d) is a one step, two step or a dual rate ageing operation to a final hold temperature between 100°C and 220°C and where the total ageing cycle is performed in a time span of between 3 and 24 hours.

11. Method according to claim 1, **characterised in that** the average grain size according to process step c) is 70 μm or less.

Patentansprüche

1. Verfahren zur Herstellung von extrudierten Profilen, die mit hochglänzenden Oberflächen anodisiert werden können, wobei die Profile aus aushärtbarer, nach Kaltumformung rekristallisierbarer Aluminiumlegierung extrudiert werden können, wobei die Legierung zuerst zu Extrusionsknüppeln gegossen wird, wobei die Knüppel bei einer Haltetemperatur zwischen 480 °C und 620 °C homogenisiert und bei dieser Temperatur 0-12 Stunden lang ausgeglichen werden, woraufhin die Knüppel Kühlung von der Homogenisierungstemperatur mit einer Rate von 150 °C/h oder schneller unterworfen werden,

a) die Knüppel auf eine Temperatur zwischen 400 und 540 °C vorerhitzt und extrudiert und schnell auf Raumtemperatur heruntergekühlt werden,
b) Umformen des Profils um mehr als 10 % durch einen Kaltwalzvorgang, woraufhin c), das Profil in einer Erhitzungszeit von maximal zwei Minuten auf eine Temperatur zwischen 400 - 530 °C schnellausgeglüht und bei dieser Temperatur nicht länger als 5 Minuten lang gehalten wird, um eine durchschnittliche Korngröße von etwa 100 μm oder weniger zu erhalten, und daraufhin abgeschreckt wird und
d) das Profil schließlich gealtert wird,
wobei
die Legierung eine 7xxx-Legierung ist, die, in Gew. -% :

Si: 0,00-0,30
Mg: 0,50-2,00
Fe: 0,00-0,15
Cu: 0,00-0,30
Mn: 0,00-0,20
Cr: 0,00-0,10
Zr: 0,00-0,20
Sc: 0,00-0,10
Zn: 3,00-7,00
Ti: 0,00-0,05 und
einschließlich zufälliger Verunreinigungen und den Rest als Al enthält.

2. Verfahren nach Anspruch 1, **dadurch gekennzeichnet, dass** das Profil zwischen Schritt c) und d) ferner einem Kaltumformungsvorgang, bevorzugt durch Strecken zum Entfernen restlicher Spannungen von Kühlen unterworfen wird.

3. Verfahren nach Anspruch 1 **dadurch gekennzeichnet, dass** das Profil zwischen Schritt c) und d) zu Rohlingen geschnitten wird, die zu einer Gestalt kaltgeformt werden, die Material und maschinelle Bearbeitungszeit spart, um das Endprodukt herzustellen.

4. Verfahren nach Anspruch 1,
dadurch gekennzeichnet, dass
das Profil Schritt b) entsprechend mehr als 20 % umgeformt wird.

5. Verfahren nach Anspruch 1,
dadurch gekennzeichnet, dass das Profil Schritt b) entsprechend bevorzugt 30 bis 50 % umgeformt wird.

6. Verfahren nach Anspruch 1,
dadurch gekennzeichnet, dass das Profil Schritt c) entsprechend in einer Erhitzungszeit von maximal 20 Sekunden auf eine Temperatur zwischen 400 - 530 °C schnellausgeglüht und bei dieser Temperatur nicht länger als 1 Minute gehalten wird.

7. Verfahren nach Anspruch 1,
dadurch gekennzeichnet, dass
das Schnellausglüherhitzen Schritt c) entsprechend durch Induktionserhitzen des Profils erhalten wird.

8. Verfahren nach Anspruch 1,
dadurch gekennzeichnet, dass das Schnellausglüherhitzen Schritt c) entsprechend durch Unterwerfen des Profils einem Salzbad- oder anderen Konvektions- oder
Strahlungserhitzungsmittel, das hohe Erhitzungsraten bereitstellt, erhalten wird.

9. Verfahren nach Anspruch 1,
dadurch gekennzeichnet, dass die Legierung ohne Verwendung von Kornfeiner, mit Ausnahme des Anfahrens des Gießvorgangs, gegossen wird.

10. Verfahren nach Anspruch 1,
dadurch gekennzeichnet, dass die Alterung, Schritt d), ein Einschritt-, Zweischritt- oder Dualratealterungsvorgang auf eine endgültige Haltetemperatur zwischen 100 °C und 220 °C ist und wobei der gesamte Alterungszyklus innerhalb einer Zeitspanne zwischen 3 und 24 Stunden ausgeführt wird.

11. Verfahren nach Anspruch 1,
dadurch gekennzeichnet, dass die durchschnittliche Korngröße Verfahrensschritt c) entsprechend 70 μm oder weniger beträgt.

Revendications

1. Procédé de fabrication de profilés extrudés qui peuvent être anodisés avec des surfaces à haut brillant, les profils étant extrudés à partir d'un alliage en aluminium durcissable au vieillissement qui peut être recristallisé après déformation à froid, où l'alliage est initialement moulé en billettes d'extrusion, où les billettes sont homogénéisées à une température de maintien comprise entre 480 °C et 620 °C et trempées à cette température durant 0 à 12 heures, où après que les billettes sont soumises à un refroidissement à partir de la température d'homogénéisation à une vitesse de 150 °C/heure ou plus rapide,

a) les billlettes sont préchauffées à une température comprise entre 400 °C et 540 °C et extrudées et refroidies rapidement à la température ambiante,
b) la déformation du profil de plus de 10 % par une opération de roulage à froid, où après c) le profil est recuit instantanément avec un temps de chauffage d'au maximum deux minutes à une température comprise entre 400 et 530 °C et maintenu à cette température durant pas plus de 5 minutes pour obtenir une taille de grain moyenne d'environ 100 μm ou moins, et ensuite désactivé, et
d) le profil est finalement vieilli,
l'alliage étant un alliage 7xxx contenant en % en poids :

Si : 0,00 à 0,30
Mg : 0,50 à 2,00
Fe : 0,00 à 0,15
Cu : 0,00 à 0,30
Mn : 0,00 à 0,20
Cr : 0,00 à 0,10
Zr : 0,00 à 0,20
Sc : 0,00 à 0,10
Zn : 3,00 à 7,00
Ti : 0,00 à 0,05, et
comprenant les impuretés secondaires et le reste Al.

2. Procédé selon la revendication 1,
caractérisé en ce que le profil entre l'étape c) et d) est en outre soumis à une opération de déformation à froid, de préférence par étirement pour enlever les stresses résiduels du refroidissement.

3. Procédé selon la revendication 1,
caractérisé en ce que le profil entre l'étape c) et d) est découpé en blancs qui est formé à froid en une forme qui sauve le matériau et le temps de machinage pour produire le produit final.

4. Procédé selon la revendication 1,
caractérisé en ce que
le profil selon l'étape b) est déformé de plus de 20 %.

5. Procédé selon la revendication 1,
caractérisé en ce que le profil selon l'étape b) est déformé entre 30 et 50 %.

6. Procédé selon la revendication 1, **caractérisé en ce que** le profil est recuit instantanément selon l'étape c) avec un temps de chauffage d'au maximum 20 secondes à une température comprise entre 400 et 530 °C et maintenu à cette température durant pas plus d'1 minute.

7. Procédé selon la revendication 1, **caractérisé en ce que** le chauffage de recuit instantané selon l'étape c) est obtenu par chauffage par induction du profil.

8. Procédé selon la revendication 1 **caractérisé en ce que** le chauffage de recuit instantané selon l'étape c) est obtenu en soumettant le profil à un bain de sel ou un autre moyen de chauffage par convection ou rayonnement fournissant des vitesses de chauffage élevées.

9. Procédé selon la revendication 1, **caractérisé en ce que** l'alliage est moulé sans l'utilisation de raffineur de grain, sauf dans le début de l'opération de moulage.

10. Procédé selon la revendication 1, **caractérisé en ce que** dans le vieillissement, l'étape d) est en une étape, deux étapes ou une opération de vieillissement à double vitesse à une température de maintien finale comprise entre 100 °C et 220 °C et où le cycle de vieillissement total est effectué dans un laps de temps compris entre 3 heures et 24 heures.

11. Procédé selon la revendication 1, **caractérisé en ce que** la taille de grain moyenne selon l'étape c) du processus est de 70 µm ou moins.

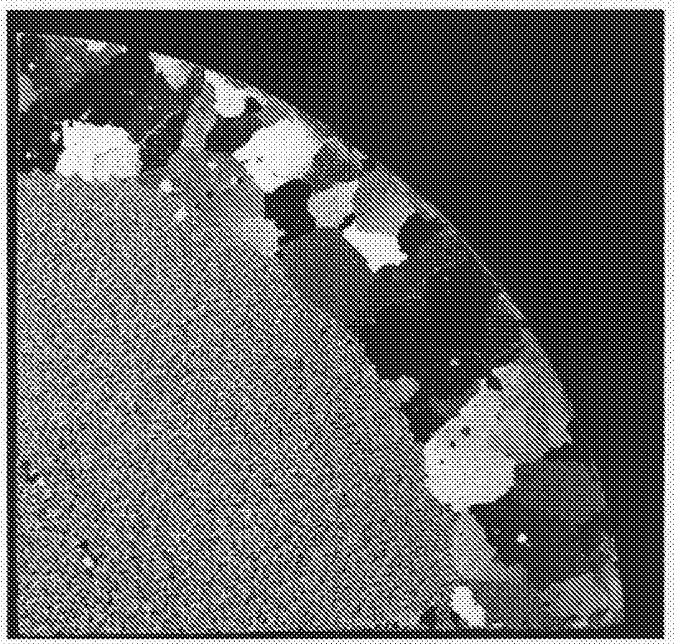


Fig. 1 Picture of a quarter of a macro etched billet slice (ø228 mm in diameter) with abnormal grains

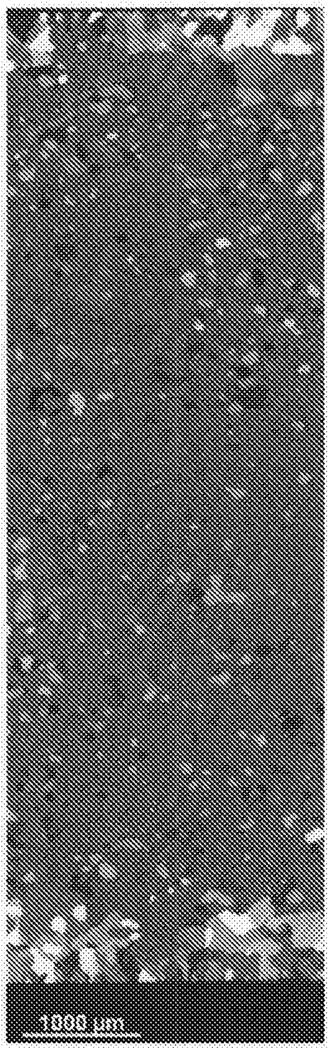


Fig. 2 Light optical micrograph showing a typical grain structure of a 6060 alloy through the thickness of a thick solid shape extruded profile. The sample is taken from a transverse cross section and is anodized and viewed in polarized light.

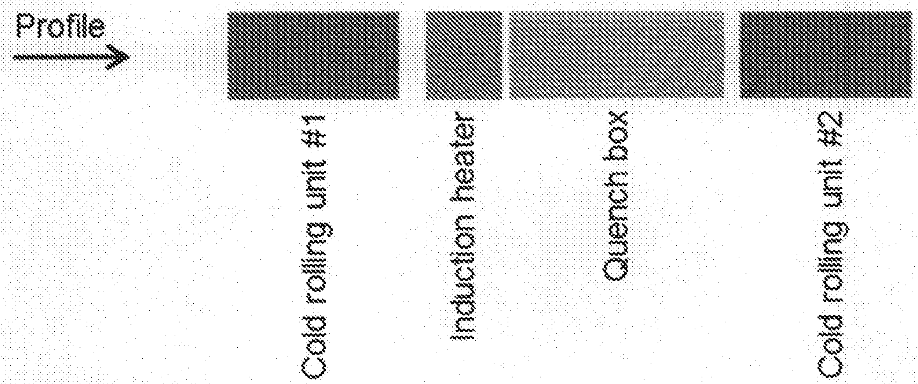


Fig. 3 A principal sketch of an industrial processing line for performing the cold rolling and the annealing process described in the present invention

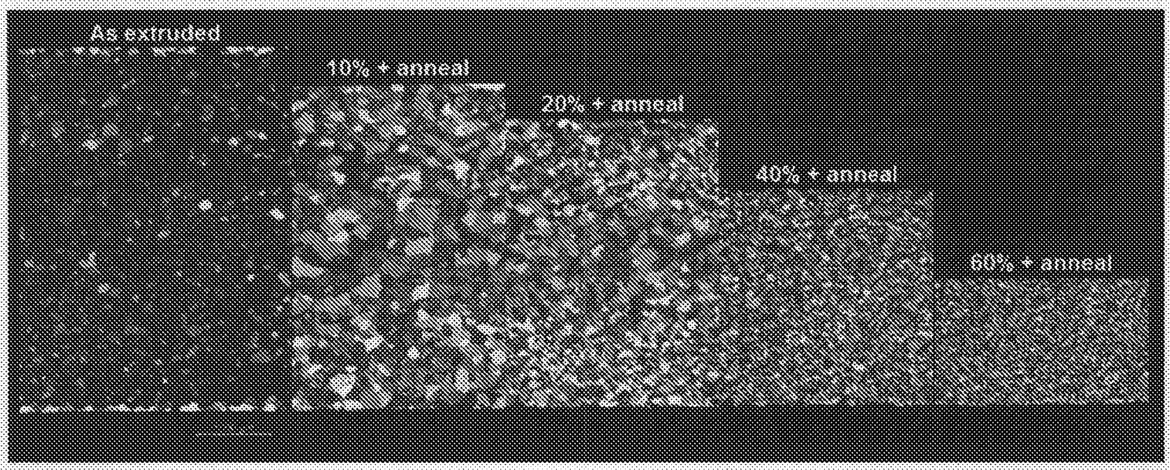


Fig. 4 Light optical micrographs of samples from example 1 showing the grain structure of a 6060 alloy in the middle of the transverse cross section for the as extruded profile and the samples that were cold rolled to give 10, 20, 40 and 60% reduction in the thickness prior to annealing. All samples are anodized and viewed in polarized light.

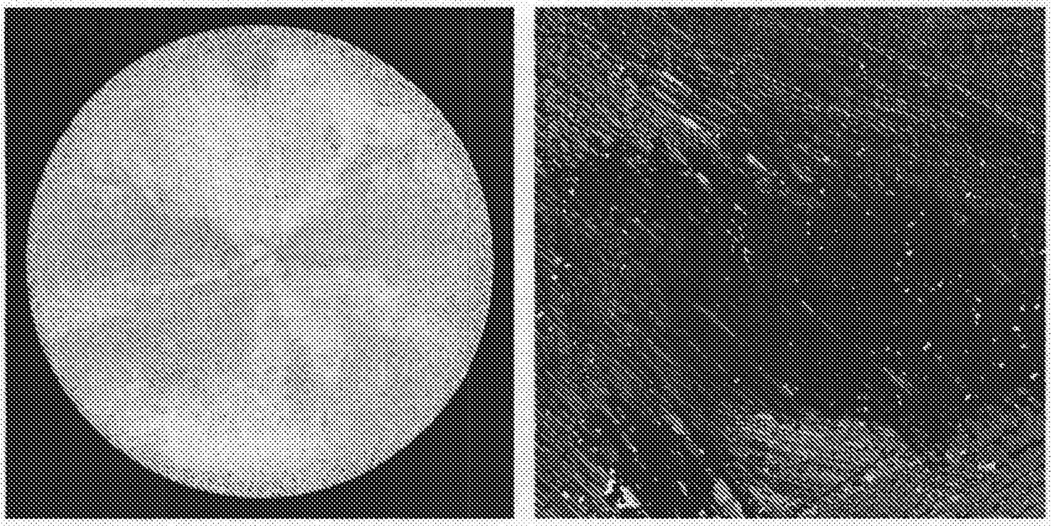


Fig. 5 Grain structure in a as cast billet (ø95 mm diameter) without grain refiner, which was used in example 2. Picture of a macro etched billet slice to the left and anodized sample viewed in polarized light in a light optical microscope to the right.

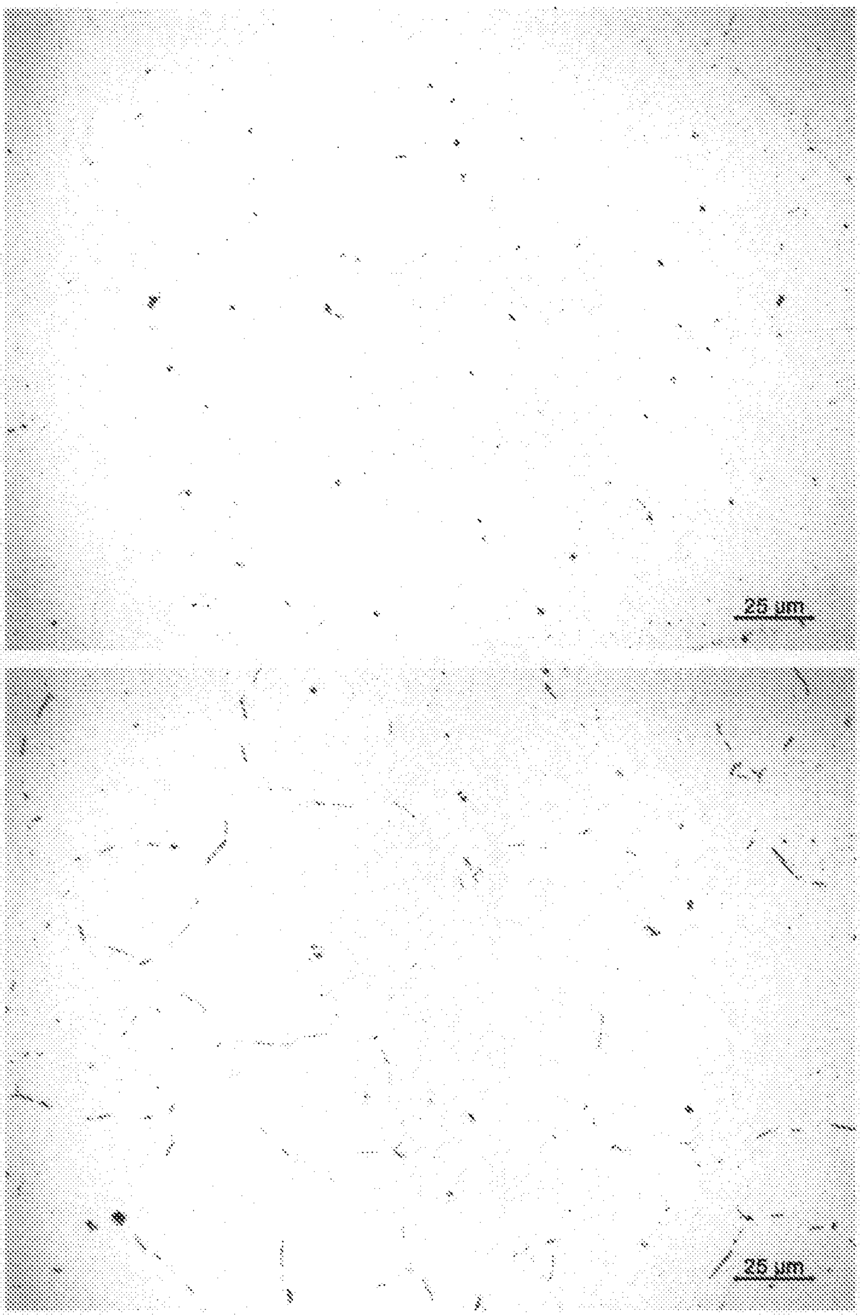


Fig. 6 Light optical micrographs showing the AlFeSi particles in a homogenized billet cast without grain refiner (upper) and in a homogenized billet cast with grain refiner (lower). The position of the samples in the billet is approximately half radius.

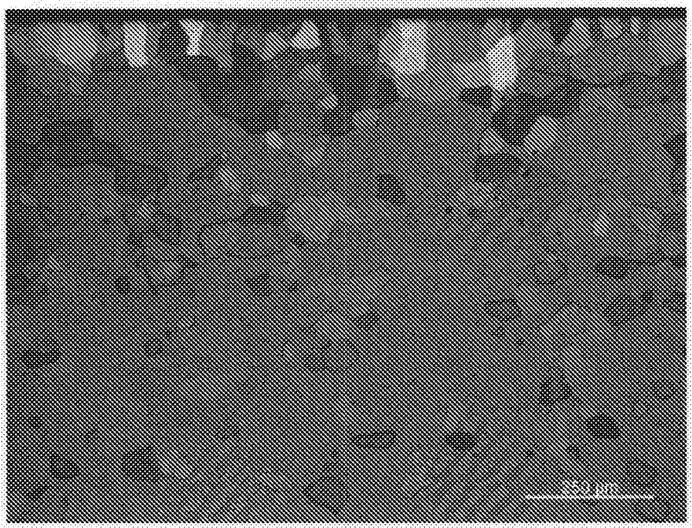


Fig. 7 Light optical micrograph of a transverse cross section of an as extruded sample in example 2, s
howing the grain structure close to the surface. Anodized and viewed in polarized light.

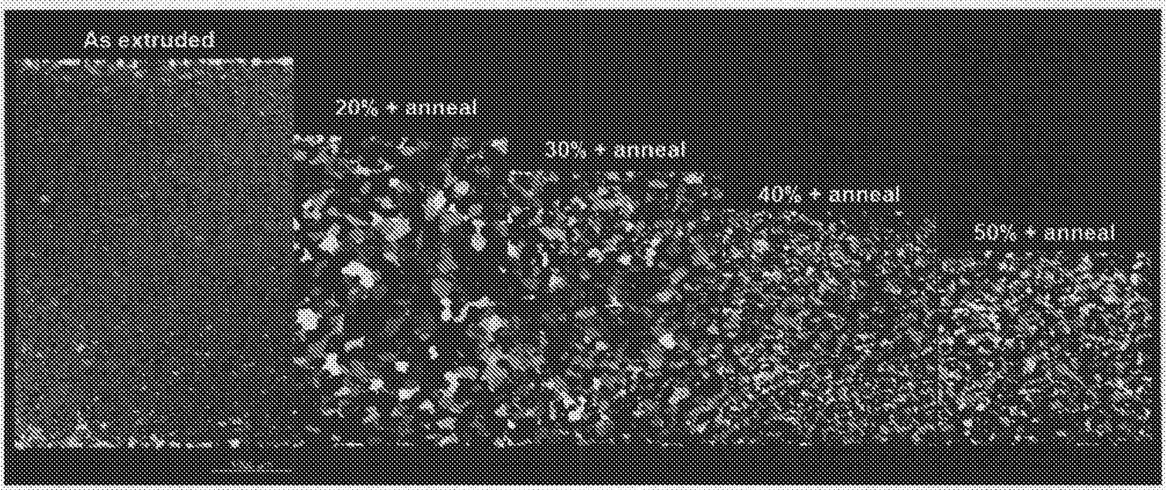
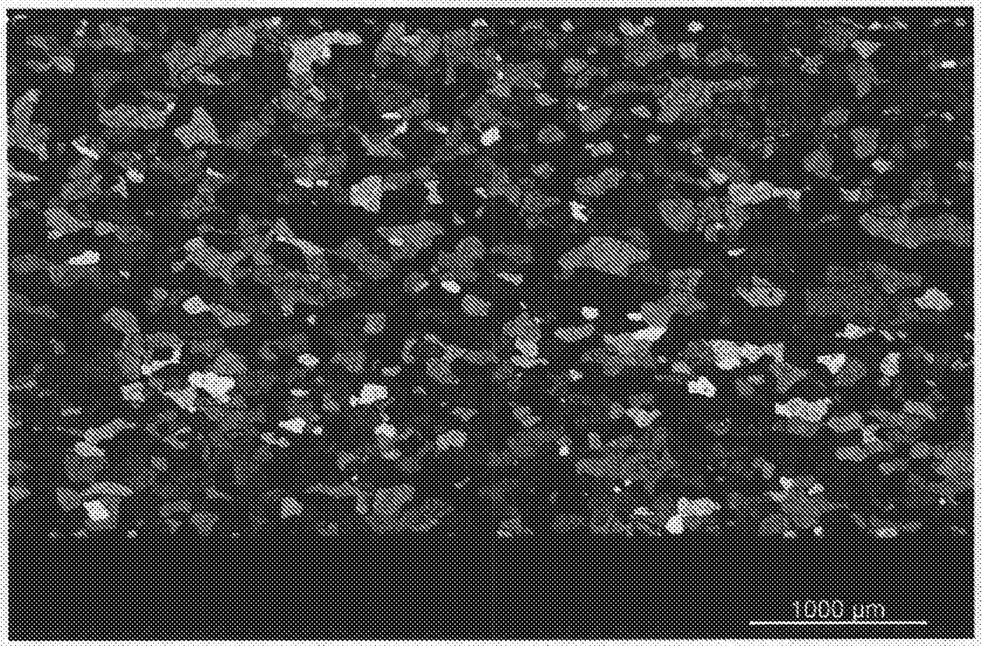


Fig. 8 Light optical micrographs of samples from example 2, showing the grain structure in the middle of the transverse cross section for the as extruded profile and the samples that were cold rolled to give 20, 30, 40 and 50% reduction in the thickness prior to annealing. All samples are anodized and viewed in polarized light.



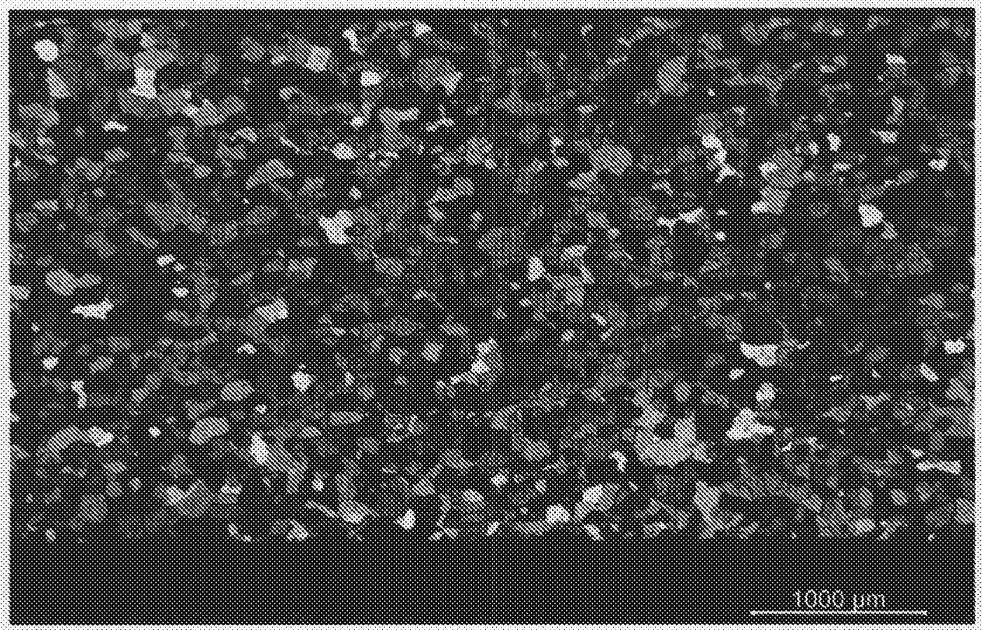


Fig. 9 Light optical micrographs of samples from example 2, showing the grain structure in the middle of the transverse cross section for samples that were cold rolled to 40% reduction in the thickness prior to annealing in air (upper) and in a salt bath (lower). Both samples are anodized and viewed in polarized light.

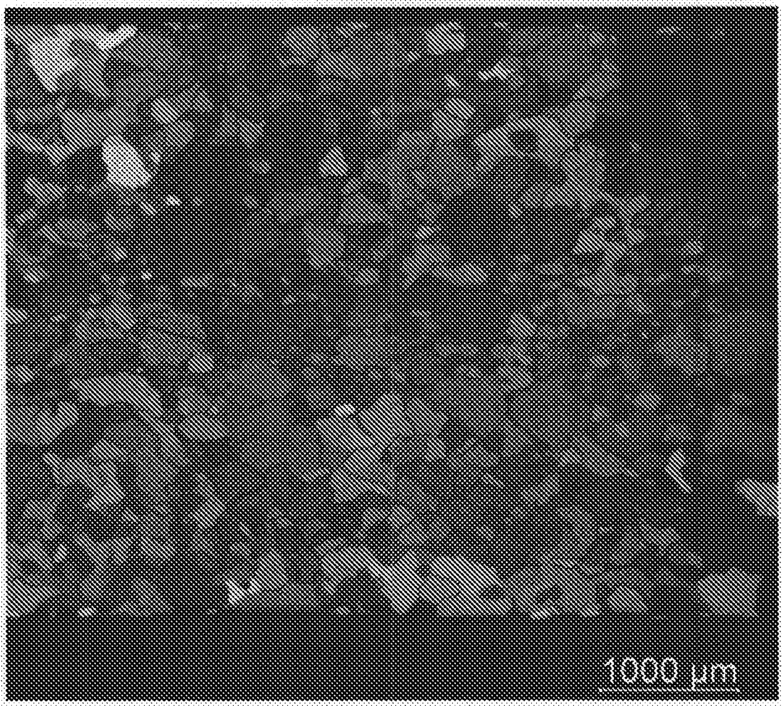


Fig. 10 Light optical micrograph of a sample of alloy 7030 from example 3, showing the grain structure through the transverse cross section of a profile that has been cold rolled to 10% reduction in thickness and subsequently flash annealed in a salt bath. The sample is anodized and viewed in polarized light.

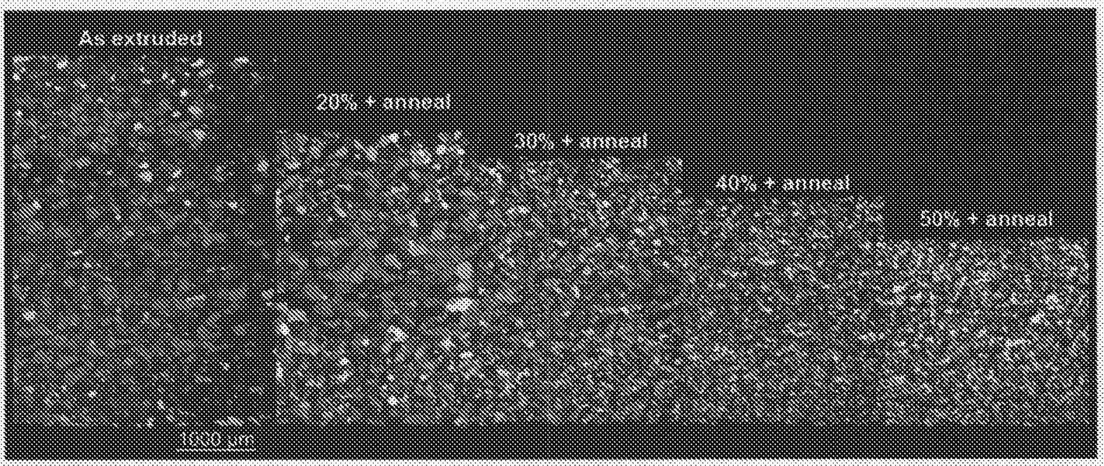


Fig. 11 Light optical micrographs of samples of alloy 7030 from example 3, showing the grain structure in the middle of the transverse cross section for the as extruded profile and the samples that were cold rolled to give 20, 30, 40 and 50% reduction in the thickness prior to annealing. All samples are anodized and viewed in polarized light.

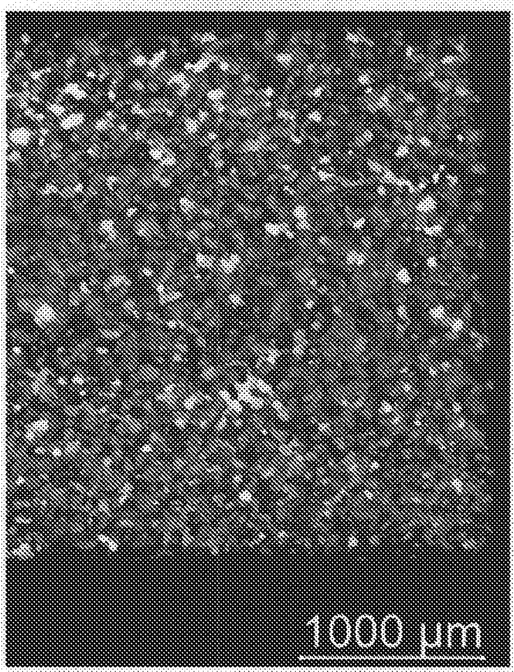


Fig. 12 The grain structure towards one end of the transverse profile cross section of a 40% cold rolled and annealed sample of alloy 7030. The sample is anodized and viewed in polarized light.

REFERENCES CITED IN THE DESCRIPTION

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