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INFLUENCE OF THE HOT-ROLLING TECHNIQUE FOR EN AW-8021B ALUMINIUM ALLOY ON THE MICROSTRUCTURAL PROPERTIES OF A COLD-ROLLED FOIL

VPLIV IZBIRE NAČINA TOPLEGA VALJANJA ALUMINIJEVE ZLITINE EN AW-8021B NA MIKROSTRUKTURNE LASTNOSTI HLADNO VALJANE FOLIJE

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In the whole manufacturing chain of aluminium products, hot rolling significantly impacts the obtaining of favourable microstructures and desired mechanical properties of final products. The determination of crucial differences between the reverse hot rolling on a single-stand mill and the tandem hot rolling on a tandem-stand mill presented a major challenge. Besides the grain-size distribution in the microstructure's cross-section, the crystallographic textures of hot-rolled strips were also determined and compared. The alternating band areas of a coarse-grained microstructure and fine-grained microstructure due to reverse hot rolling and, especially, the appearance of extremely fine grains on the surfaces present limitations compared to the tandem hot rolling. For subsequently cold-rolled foils, classical mechanical properties were measured. Besides, the usefulness of EN AW-8021B foils with a thickness of 60 µm for pharmaceutical-packaging applications was tested with a burst test. A minor but important difference of 1 % in the elongation is shown for the convex height increased by 1 mm.

Keywords: aluminium alloys, hot rolling, microstructure, EBSD, burst test

V celotni verigi proizvodnje aluminijevih izdelkov ima toplo valjanje še posebej velik vpliv na ustvarjanje ugodne mikrostrukture in želene mehanske lastnosti končnih izdelkov. Osrednji izziv je predstavljalo določevanje ključnih razlik med primerjanima reverzirnim in tandemskim toplim valjanjem. Ob razporeditvi povprečne velikosti kristalnih zrn po prečnem preseku, se je določila in primerjala tudi kristalografska tekstura toplo valjanih trakov. Spreminjajoča področja fino in grobo zrnate mikrostrukture po prečnem preseku, predvsem pa izjemno drobno zrnata mikrostruktura na obeh robovih reverzirno toplo valjanih vzorcev, predstavlja slabše izhodišče za hladno valjanje v primerjavi z vzorci tandemskega toplega valjanja. Za nadaljnje hladno valjanje folije, pa so bili izvedeni še natezni preizkusi. Ob tem je bila, s preizkusom napihovanja oz. z razpočnim preizkusom, testirana tudi uporabnost folij iz EN AW-8021B z debelino 60 µm za embalažo v farmacevtskih aplikacijah. Precej majhna (1 %), a pomembna razlika je v raztezku, in sicer pri razliki 1 mm konveksne višine.

Ključne besede: aluminijeve zlitine, toplo valjanje, mikrostruktura, EBSD, razpočni preizkus

1 INTRODUCTION

During the manufacturing, a final rolled aluminium product undergoes different metallurgical processes with mechanical, technological, thermal and deformation effects.[1] The first step of the process is most often casting. That is either semi-continuous casting (slab)[2,3] or rolled casting (strip).[4,5] Transforming an aluminium cast slab with hot rolling to a flat sheet, which is then the starting material (stock) for cold rolling, is one of the most common and cost-effective techniques.[6] With the favourable mechanical properties, cold-rolled aluminium products are used in the automotive, transport and packaging industry. The production of electrical conductors is a major consumer of 8xxx rolled aluminium alloys[7] while the EN AW-8021B aluminium alloy foil is an exception with respect to pharmaceutical packaging needs.

Aluminium hot-rolling mills typically have different arrangements. The single-stand mill or the tandem-stand mill, where the latter can have one or more finishing mills, are the most common configurations.[7] Considering the amount and variety of products as the major selection criteria, the produced material characteristics (microstructure, texture and mechanical properties) resulting from different mill arrangements remain widely unresearched. Ahmed et al.[8] generally divide aluminium hot rolling into four stages. These include pre-heating, roughing, finishing and coiling. The microstructure and mechanical properties of a material change during hot rolling due to its heat transfer, thermomechanical state, composition, strip speed and thickness, and reduction percent.[9] As summarised in the literature[8,9] there are more hot-rolling production parameters with different intensities of influences. A quantitative analysis of four process parameters during hot rolling of aluminium alloy AA 1100 was done by Duan and Sheppard.[10] The authors

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Table 1: Chemical composition of investigated aluminium rolled products in *w*/%

Process of hot rolling	Sample designation	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Al
Reverse	HR_{rev}	0.12	1.50	0.02	<0.01	<0.01	<0.01	<0.01	0.03	Bal.
Tandem	HR_{tan}									

concluded that the slab temperature causes a 64.2 % change in the subgrain size (consequently also the grain size), followed by the change in the reduction by 29.6 %, roller's speed by 5.0 % and temperature of rollers by only 1.2 %. Venkateswarlu et al.[11] present, in more detail, the grain refining from 15.4 µm at a rolling temperature of 400 °C up to 10.5 µm at 200 °C and a reduction of 80 %.

The purpose of hot rolling aluminium alloys is primarily to produce desired mechanical properties, which also strongly depend on the associated microstructures. Butt et al.[12] produced an AA 1050 foil (a thickness of 100 µm in the H14 condition) with a tensile strength of 120 MPa and yield strength of around 105 MPa. Somewhat higher tensile strength (150 MPa) and yield strength (130 MPa) were reached by Can et al.[13] with an AA 8079 foil of 6.35 µm. The elongation of this foil was only 4.5 %. Quality determination of aluminium foils exceeds the classical mechanical tests. Specific investigations using the Erichsen cupping test[14] or the plastic-strain-ratio test,[15] where the determination of the formability or anisotropy is required, are part of rolled-product studies and quality control. The burst test is versatile and valuable, especially for the foils (pharmaceutical and packaging applications) but rarely a part of investigations.

The aim of this work was to compare different hot-rolled strips of the EN AW-8021B aluminium alloy. Microstructural and texture variances between reverse- and tandem-hot-rolled materials were investigated. Any significant influence of hot rolling was directly correlated to the mechanical properties of foils with a thickness of 60 µm that were later cold rolled. Relatively similar results for the tensile test were increased with the convex height and pressure of the burst test. The values obtained indicated the limits to the advancement of the foil properties for pharmaceutical-packaging applications.

2 MATERIAL AND METHODS

Aluminium alloy EN AW-8021B was industrially shaped into slabs via direct-chill casting (DC). The chemical composition of both subsequently rolled samples is presented in **Table 1**. The slabs were hot rolled on a single-stand mill (**Figure 1a**) or a tandem-stand mill with one reverse-roughing mill and four finishing mills (**Figure 1b**) to the defined thickness. During the rolling on the reverse single-stand mill, the workpiece is reverse rough rolled to the suitable thickness for coiling. When an even lower thickness of the rolled product must be achieved, the uncoiling system is used, finishing the rolling with some additional passes (on the same rolling mill) where the coiling is always present. On the tandem-hot-rolling mill, the workpiece is reverse-rough rolled (the same as with the previous system) and finished on the four tandem finishing mills where the coiling of a strip with the desired thickness takes place. Further, cold rolling was executed obtaining the foil's thickness of 60 µm where the treatment solution was performed for the intermediate thickness of the rolled product. The same cold-rolling schedule was performed for reverse-hot-rolled sample HR_{rev} and tandem-hot-rolled sample HR_{tan}.

The samples of rolled products were metallographically prepared by grinding and polishing for the microstructural and texture analyses with a light microscope (LM) and scanning electron microscope (SEM). They were electrolytically etched with Barker's reagent for the observation under polarised light (in LM). The grain-size determination was performed in accordance to the ASTM E112 – 13 standard. For the texture studies, electron backscatter diffraction (EBSD) coupled with SEM was performed. The recorded EBSD mappings were further converted into pole figures and orientations at the upper and bottom sample surfaces and at the centre were discussed.

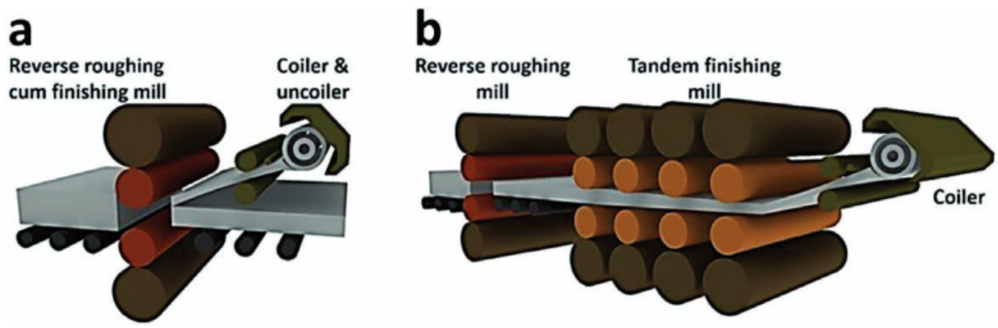

Figure 1: Schematic presentation of aluminium hot-rolling mills: a) single-stand mill, b) tandem-stand mill (1+4 tandem)

Mechanical properties of the foils (tensile strength, yield strength and elongation) were determined according to SIST EN 546-2:2007. The results were presented as the average values measured in the rolling and transverse directions. Like the tensile test, the burst test was also repeated several times for both rolled foils. The mentioned test was performed at different positions (edges, centre) covering a large rolled-product area.

3 RESULTS AND DISCUSSION

3.1 Hot-rolled strips

The microstructure of hot-rolled products has, from the point of view of subsequent cold rolling, supreme importance.[16] The temperatures that are too high at the end of hot rolling result in grains that are also too large. The coarse-grained microstructure is problematic for the subsequent cold rolling of the sample to a lower thickness. The above phenomenon directly causes a decline in the mechanical properties, especially the foil's elongation values that are too low. Besides the temperature intervals, the impact of the whole thermomechanical process (the chosen hot-rolling system) has a significant meaning for the microstructure grain size, formation and homogeneity. During reverse hot rolling (**Figure 2a**), the areas with different grain sizes in the HR_{rev} microstructure cross-section are changed several times. From the upper surface, where the extremely fine grains are found, some larger and recrystallized grains follow to the centre of the cross-section. Also, in the centre, similar, larger grains are observed. The fine-grained microstructure, similar to that at the edge of the upper surface, is between areas with coarser grains. In the cross-section, between the centre and the bottom surface, mirrored sequences, like the ones on the upper part, with fine and coarse microstructure areas were created. On the observed microstructural bands, where the fine-grained microstructure changes into the coarse-grained microstructure and vice versa, the heterogeneity is increased, making it challenging to provide a homogeneous grain-size distribution of the product to be further cold rolled. The fine-grained microstructures on both surfaces present additional difficulties for cold rolling. Samples of HR_{tan} are prepared for cold rolling in a different way as more recrystallized and larger grains are on the upper and bottom surfaces (**Figure 2b**). In the centre of the HR_{tan} cross-section, longitudinal and deformed grains are created. In this microstructure, no bands with alternating fine and coarse grain areas are observed. This is the primary indicator for a more homogeneous microstructure.

The above microstructural differences are also confirmed with the determined and calculated average grain-size values for the three positions in the cross-sections (**Figure 3**). The highest average grain size of (48 ± 3) µm was found on the bottom surface of the HR_{tan} sample. At the same position, but on the other sample (HR_{rev}), the smallest average grain-size value of (26 ± 2) µm was determined. For the HR_{rev} sample, the average grain-size value increased to (43 ± 4) µm in the direction of the centre of the cross-section and, again, it became more refined (26 ± 3 µm) on the upper surface. Compared to the above HR_{rev} sample, different sequences were found in the HR_{tan} samples, where the grain size on the upper surface was (45 ± 4) µm. The grain-size refinement for the HR_{tan} samples was observed in the centre, where the average grain size was (29 ± 2) µm. Thus, the average grain-size values throughout the cross-section differ greatly in both microstructures, which is a significant indicator of a heterogeneous microstructure. Despite this, in the case of cold rolling, the microstructure of the HR_{tan} sample is more

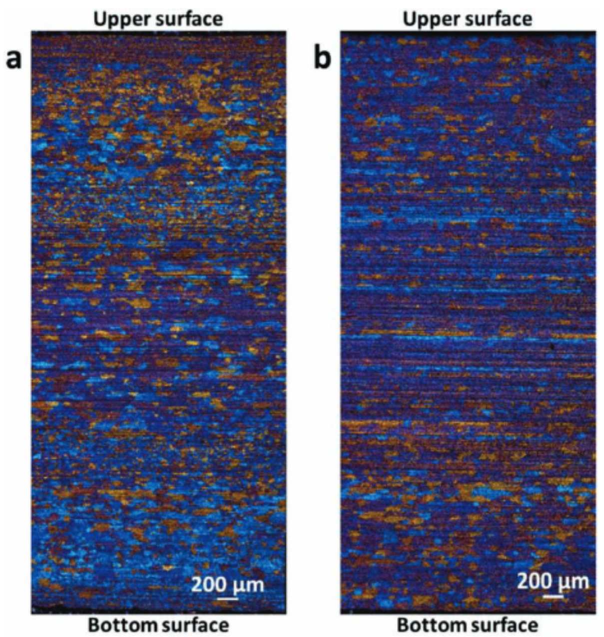

Figure 2: LM microstructure of the cross-section of a hot-rolled strip: a) reverse rolling, b) tandem rolling

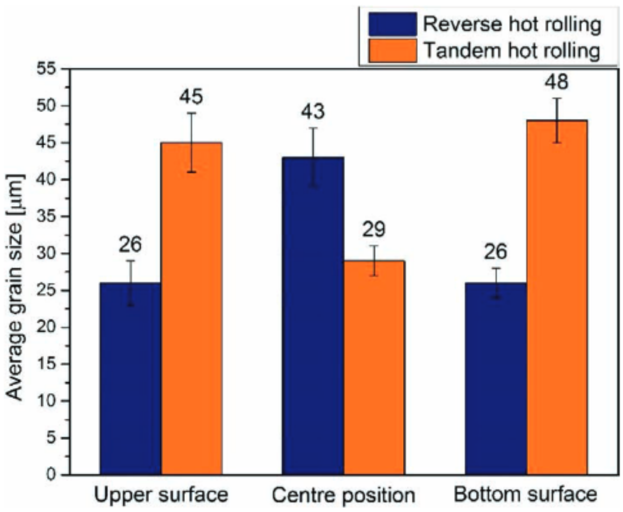

Figure 3: Average grain sizes of hot-rolled strips measured at different positions of the cross-sections

appropriate than that of the HR_{rev} sample due to its larger grains on the surface.

Similarly to the LM analysis of the cross-sections covering the area from the upper to the bottom surface, the SEM-EBSD mappings were performed for the HR_{rev} (**Figure 4a**) and HR_{tan} (**Figure 4b**) samples. Once again, completely different sequences of the average grain sizes of the two materials were presented with inverse pole figures in the Z direction (IPF-Z). Moreover, the pole figures with their intensities coincided with the average grain sizes. In all the cases of a large average grain size, the intensity of the observed texture is higher. In the centre of both samples, the classical rolling-texture connection between different rolling-texture components, also known as the β-fibre, is recognisable. In addition to this fibre, which is predominant, a cubic texture component is present on pole figures. A much more cubic texture component is observed in the centre of the HR_{rev} sample. At the same time, this inhibits a stronger formation of the β-fibre. Comparing the textures of HR_{rev} and HR_{tan} samples' upper and bottom surfaces, more pronounced differences are observed, similar to the ones previously found for the centre positions. Instead of the β-fibre, which is always present along stronger deformations,[17] the well-recognised and predominant α-fibre is created on both surfaces of HR_{rev}. The α-fibre connects fewer texture components than the β-fibre, and its creation is conditioned with lower deformations.[18] An almost entirely recrystallized texture with the most predominant cubic texture component is observed on the upper and bottom surface of the HR_{tan} sample. Besides the average grain-size difference (smaller grains) and the texture that is less recrystallized and based on the more rolling-orientated α-fibre of the HR_{rev} sample, it is once more important to emphasise that such a material represents a less favourable initial condition for cold rolling.

3.2 Cold-rolled foils

Some microstructural differences observed in hot-rolled strips were transferred from cold rolling to the final foil thickness of 60 µm. Individual large grains are especially outstanding in **Figure 5a**, which presents the microstructure (IPF-Z) of the cold-rolled foil obtained from the HR_{rev} sample. The average grain size for the above microstructure is (12 ± 1) µm. In **Figure 5b**, the microstructure with IPF-Z of the cold-rolled foil made from the HR_{tan} sample is presented. A more homogeneous microstructure with an average grain size of (9 ± 1) µm is the result of better initial conditions for cold rolling created with tandem hot rolling. Despite the very small but crucial differences in the average grain size of the two foils compared on such a low scale (60 µm foil thickness), each deviation in the microstructure has a significant impact, consequently reducing the mechanical properties that are desired for a certain application.[19]

The above average grain sizes are further highlighted with a presentation of the whole-grain size distribution in **Figure 6**. It is clearly shown that in the microstructure of

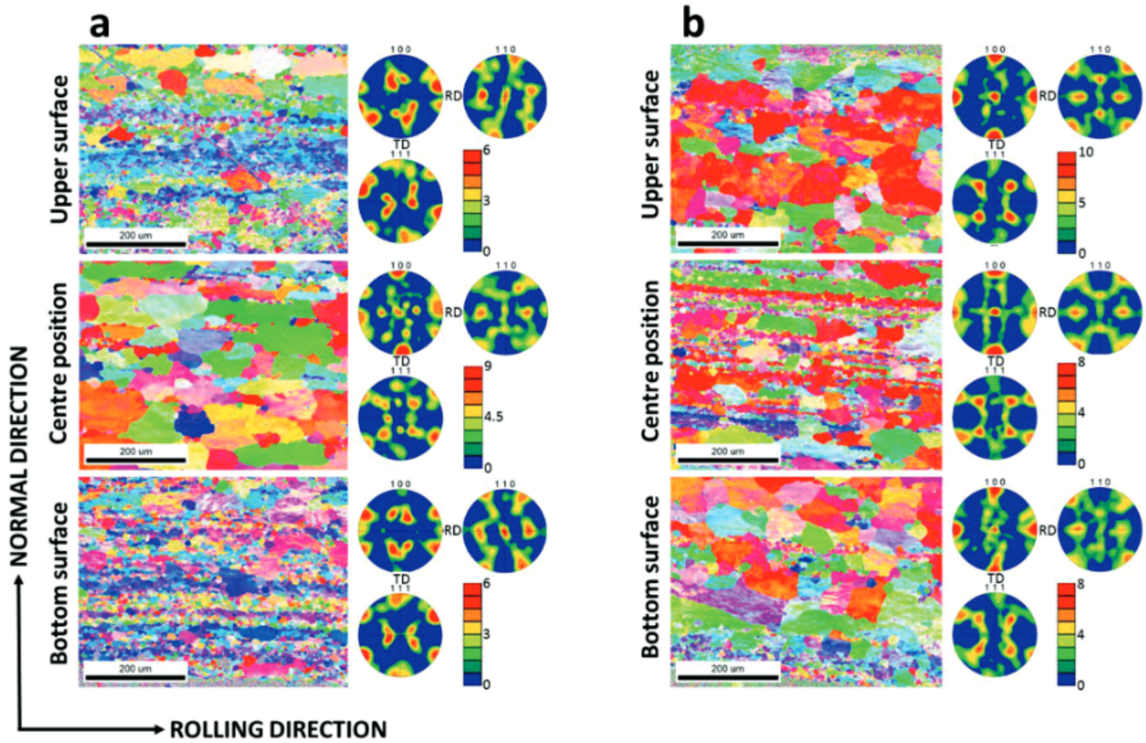

Figure 4: Crystallographic textures of the upper surface, centre and bottom surface of hot-rolled strips presented with IPF-Z and pole figures: a) reverse rolling, b) tandem rolling

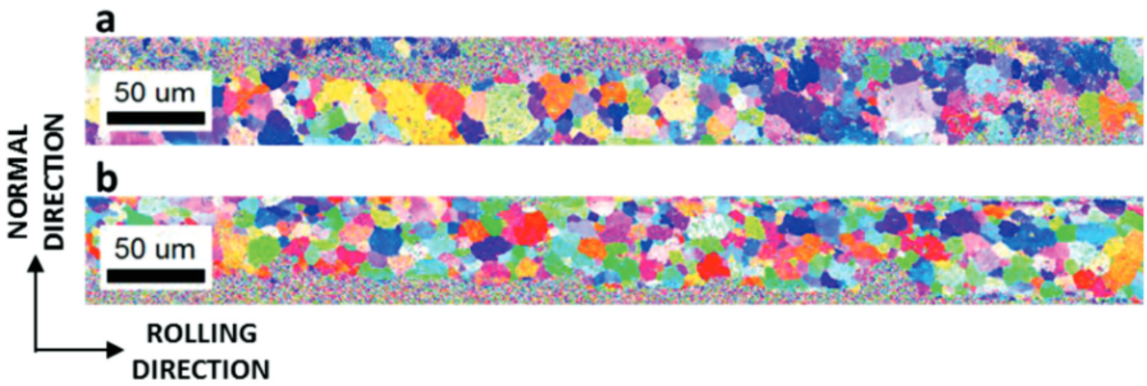

Figure 5: SEM-EBSD microstructure cross-sections of cold-rolled foils: a) reverse rolling, b) tandem rolling

the cold-rolled foil, which was previously tandem hot rolled, the largest grains are around 17 μm. Their share is 9 %. The same percentage of much larger grains (26 μm) is observed in the microstructure of the cold-rolled foil, which was previously reverse hot rolled. In addition to this fundamental difference, the whole-grain distribution with two peaks higher than 12 % is presented for the foil rolled from the HR_{rev} sample. On the contrary, the microstructure of the cold-rolled foil made from the HR_{tan} sample has a distribution with only one peak. The presence of larger grains reduces the changes needed to achieve the desired elongations.[20]

Foil-sample surfaces were oxide polished and analysed with EBSD. **Figure 7a** presents the IPF-Z and pole figures for the cold-rolled foil previously reverse hot rolled. Besides a smaller intensity and minor differences in the presented texture components in pole figures, more significant variances are observed in the middle figure (between IPF-Z and pole figures) where only grains with the cubic-texture orientation are present. Comparing **Figures 7a** and **7b,** we find that the cold-rolled foils that were previously tandem hot rolled have a few percent smaller share of the cubic texture. Besides the smaller share of the major recrystallization texture components, a clustered distribution and band formation were observed. The latter has a direct negative impact on desired mechanical properties.[21]

Between the measured tensile and yield strength, only minor (almost insignificant) differences were noticed. The major difference was observed due to the specific burst test results where more than 1 mm difference in the convex height was recorded. Such a difference strongly impacts the foil formability, deep-drawability and elongation, which are the most important properties of foils to be used in the pharmaceutical packaging industry.[22,23] For an increase in the convex height and pressure during a burst test, the ratio between the tensile strength and yield strength is often important. With a

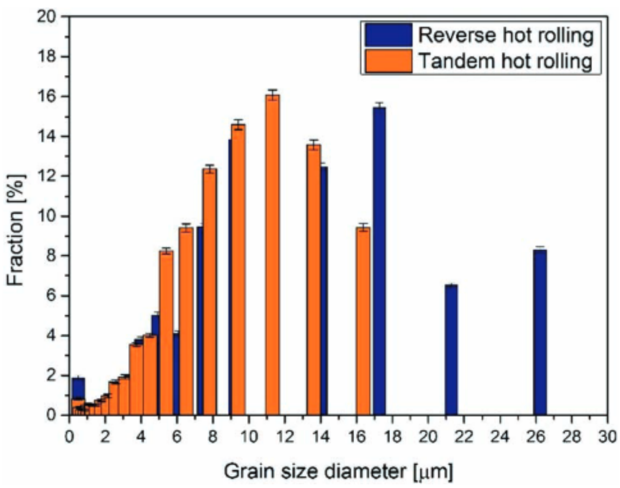

Figure 6: Correlation between fraction and grain diameter for the foils, previously reverse hot rolled or tandem rolled

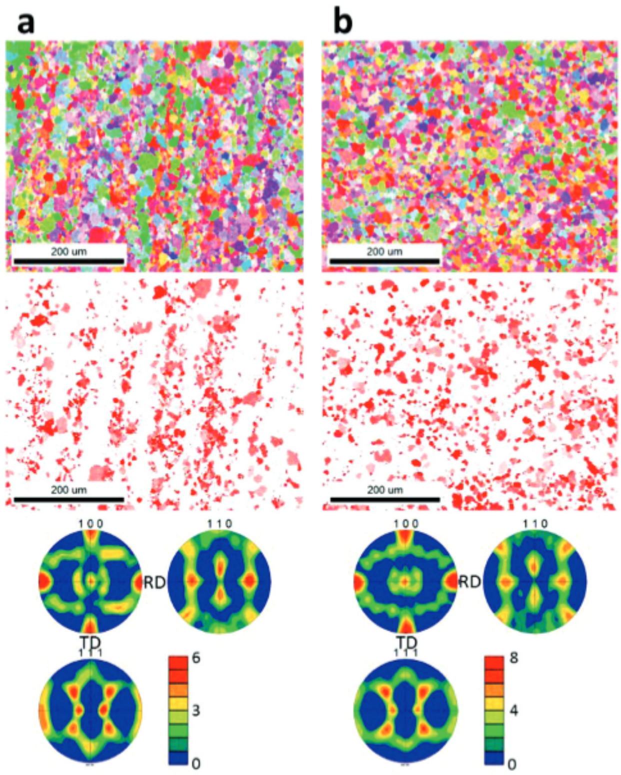

Figure 7: SEM-EBSD surface-texture presentation with IPF-Z, cube-texture component and pole figures of cold-rolled foils: a) previously reverse hot rolled, b) previously tandem hot rolled

higher ratio between these two quantities, a larger area of plastic deformation to fracture is guaranteed, consequently improving the formability of the material.

4 CONCLUSIONS

The reverse and tandem hot rolling were characterised based on the microstructure and texture of the EN AW-8021B aluminium alloy. As for the comparison of the final materials, the subsequently cold-rolled foils from the two differently rolled strips were investigated with tensile and burst tests. The effects and impacts of the chosen hot rolling system on the microstructure, texture and mechanical properties were as follows:

A transient microstructure where the coarse-grain areas alternate with fine-grain regions and vice versa is created in the cross-section of the reverse-hot-rolled sample. A much more homogeneous distribution is observed in the microstructure cross-section of the tandem-hot-rolled sample.

All the hot-rolling-related deficiencies in the microstructure and texture are also observed after cold rolling. The homogeneity in the cross-section and the recrystallization of grains, especially on the surfaces, provide better mechanical properties after cold rolling. Better material properties for subsequent cold rolling are achieved after tandem hot rolling.

The suitability of reverse hot rolling for selected foil applications is confirmed with almost the same tensile-test results as those achieved with tandem hot rolling. Nevertheless, the insufficiency of the burst-test results must be compensated with the corrections in the hot-rolling technology.

Further research is the key to obtaining a proper microstructure and mechanical properties of a reverse-hot-rolled strip, which would be more similar to the tandem-hot-rolled strip. The solution may be a decrease in the starting or finishing temperature of a workpiece. In addition, variations in the deformation (reduction) or rolling speed can also be successful for small improvements of foil properties.

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