

# Characterization of grain structure and crystallographic texture variation during the production of a Properzi semi-product

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**Abstract.** The solidification on the casting wheel is the first step of the Properzi process which is followed by rolling in several steps, in this case 11 rolling steps. The uniqueness of the technology is that the rolling occurs from 3 directions. Therefore, a completely unique texture develops in the product compared to classic rolling. This aluminium semi-product is the base product for wire drawing. The characteristic of the strand greatly influences the success of the wire drawing therefore, knowing the crystallographic texture is demanded. In this paper, the grain structure and crystallographic texture variation during the full production technology of a Properzi semi-product is characterised. The examined strand includes all the technological steps of the process (casting and the 11 rolling steps). To perform such investigation, strand the complete production must be stopped for sampling because of the closed and semi-continuous type of the process. Therefore, the presented work is unique. To characterize the semi-product, cross-sectional and longitudinal-sectional samples were prepared. Grain structure and X-ray diffraction (XRD) texture examinations were carried out on the two sample series. Based on the results it can be stated, that despite of the grain refinement, initially a columnar structure formed and a strong <100> texture developed. During rolling, the <111> fibre texture developed. After the last step, the initial <100> texture is still present besides the <111> fibre texture. Consequently, the initial conditions of the crystallization highly determine the developed texture of the semi-product.

## 1. Introduction

The mechanical properties and formability of the products and semi-products are highly determined by the crystallographic texture, therefore knowing the texture development during the technology steps helps to understand the inter process behaviour and final texture of the product.

The Properzi technology is a unique and different process compared to classic rolling. While during classic rolling the rolling occurs from 2 directions, for the Properzi process, the rolling occurs from 3 directions, where the angle between the neighboring rolls are is 120°. Due to this configuration, a unique and different crystallographic texture compared to classic rolling develops within the product. The process itself is a half-continuous, closed system. Because of that, sampling is very complicated and requires the stop of the manufacturing line. In this paper, a strand produced by Properzi process was examined after every technological step. The aim of this paper is to characterize the crystallographic texture and grain structure variation during the full Properzi production technology. To achieve this aim, XRD texture and grain structure examinations were carried out.



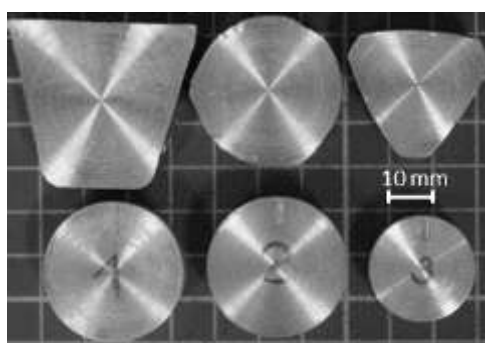
## 2. Experimental

The examined aluminium strand was produced by Properzi process. For sampling, the whole process was stopped and a section of the strand including all technological steps (casting and 11 rolling steps) was taken out. The obtained strand is shown in Fig. 1.



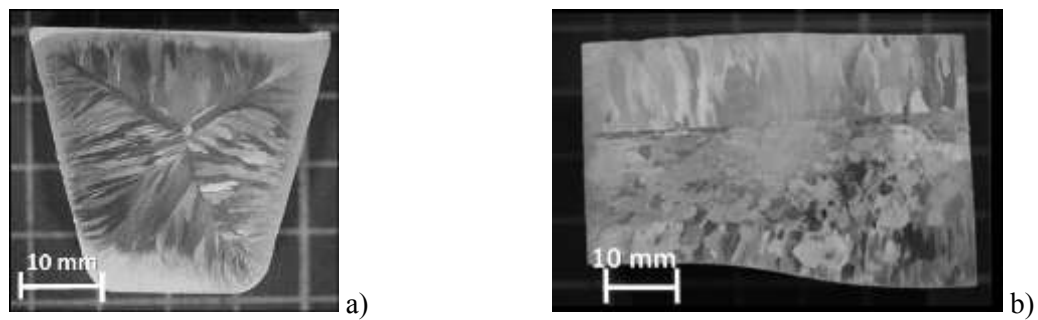
**Figure 1.** The examined strand in two pieces including all the technological steps

XRD texture measurements were carried out with a Bruker D8 Advance X-ray diffractometer equipped with an Eulerian cradle. For the measurements, circular cross-sectional samples were machined from each technological step (Fig. 2.). The first step of the texture measurement was to record the full interference function of the samples, followed by incomplete (up to 75° tilting) pole figure measurements. Afterwards, the Orientation Distribution Function (ODF) and the volume fractions of the favoured unit cell orientations were calculated. The Eulerian angles of ODF are connected to the coordinate system of classic rolling. Furthermore, the rotation axes of the Eulerian angles are bonded to the sample holder of the X-ray diffractometer. Therefore, to properly define the unit cell orientations in the Properzi coordinate system, the rotation axes and directions of the Eulerian angles had to be implemented into the coordinate system of the Properzi samples [1-4].



**Figure 2.** Cross-section samples after the 1<sup>st</sup>, 2<sup>nd</sup> and 3<sup>rd</sup> manufacturing steps prepared for XRD measurements

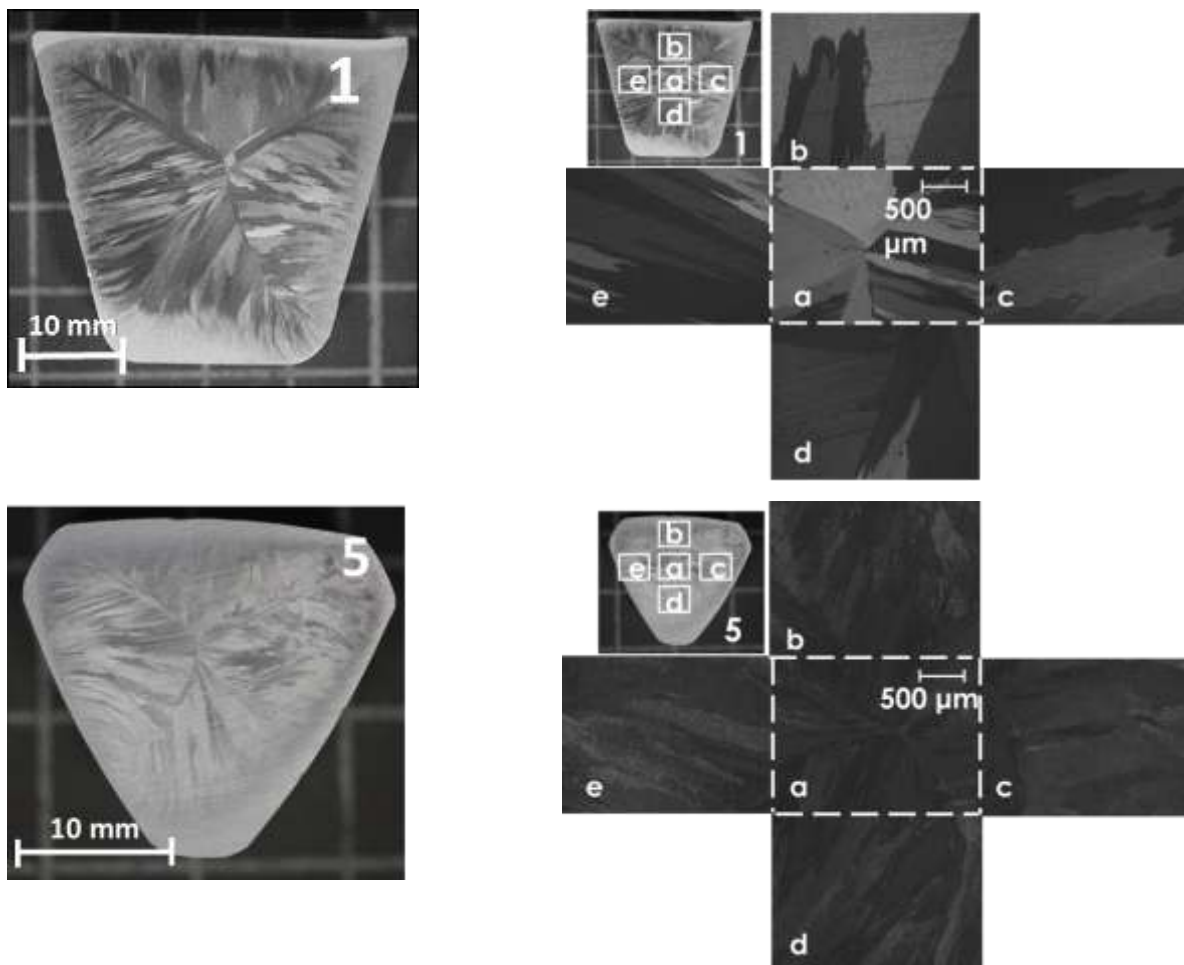
The grain structure examinations were carried out on two sample series, cross-sectional (Fig. 3a) and longitudinal-sectional (Fig. 3b). The cross-sectional samples were turned out from each technological step, meanwhile the longitudinal-sectional samples were machined out from the transitions between two subsequent steps, called transitional zones. After sample' preparations (grinding, polishing), two different etching techniques were used for both sample series. First, macro etching with caustic containing HNO<sub>3</sub>, HCL, HF and subsequently, re-preparation of the same sample with Barker etchant with caustic containing distilled water, HBF<sub>4</sub> were performed. For the examination of the grain structure for macro etching, a Canon EOS 700D + EFS 60 mm SLR camera and for Barker etching Zeiss AxioVert 100 optical microscope with polarized light were used [5-6].

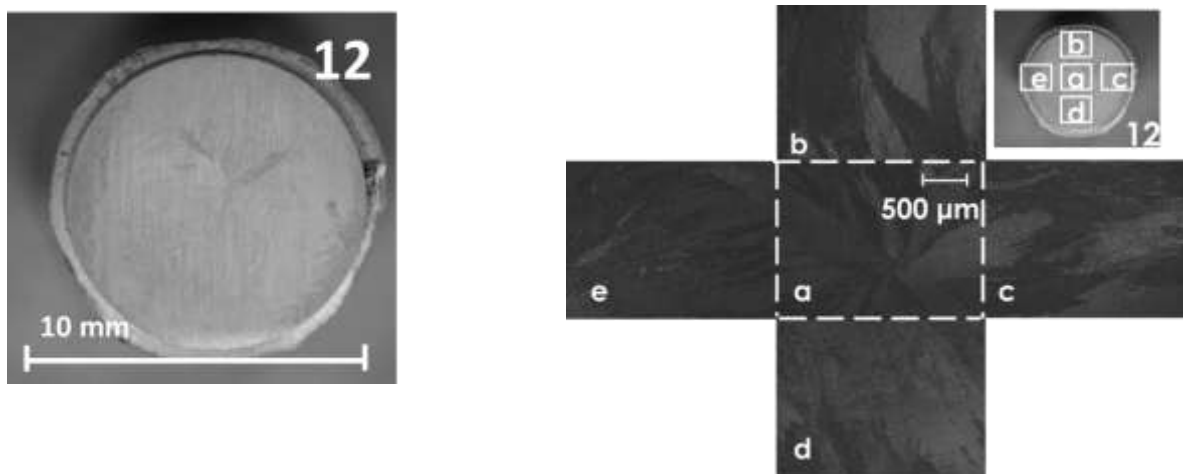


**Figure 3.** The 1<sup>st</sup> cross-sectional sample a) and the 1<sup>st</sup> longitudinal-sectional sample b)

### 3. Results and Discussion

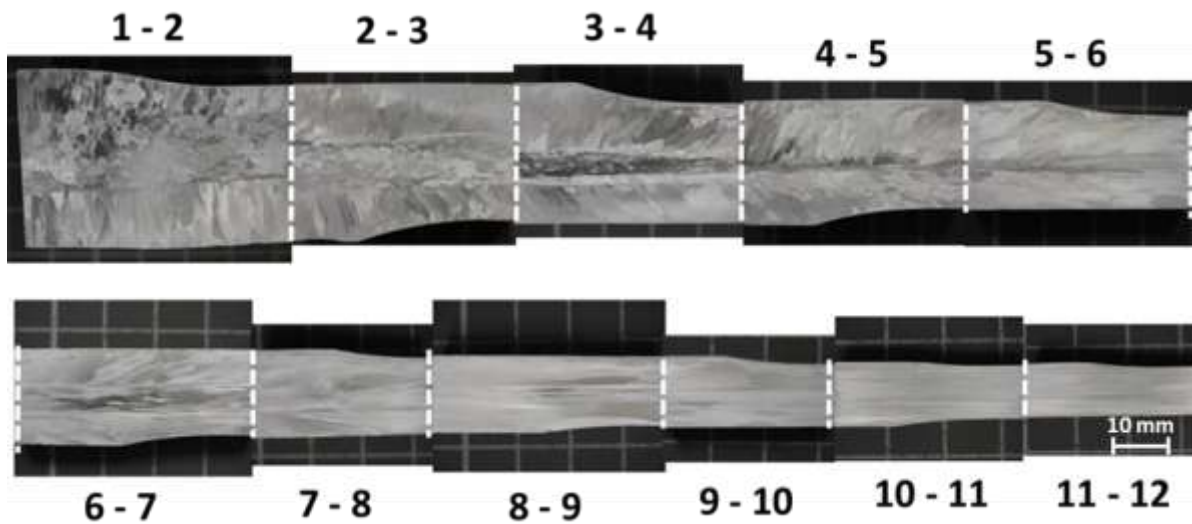
Fig. 4 shows the results of the macro and barker etched cross-sectional samples after the 1<sup>st</sup>, 5<sup>th</sup> and 12<sup>th</sup> technology steps. Due to the macro etching, the grain structure is visible for the naked eye. After each step, it can be observed that 4 zones formed according to the 4 directions of heat transfer during solidification. It can be also seen that the zones meet in the centre of the strand, furthermore, the grains formed a columnar structure within the zones despite grain refinement. As the technology proceeds the grains are more severely formed.





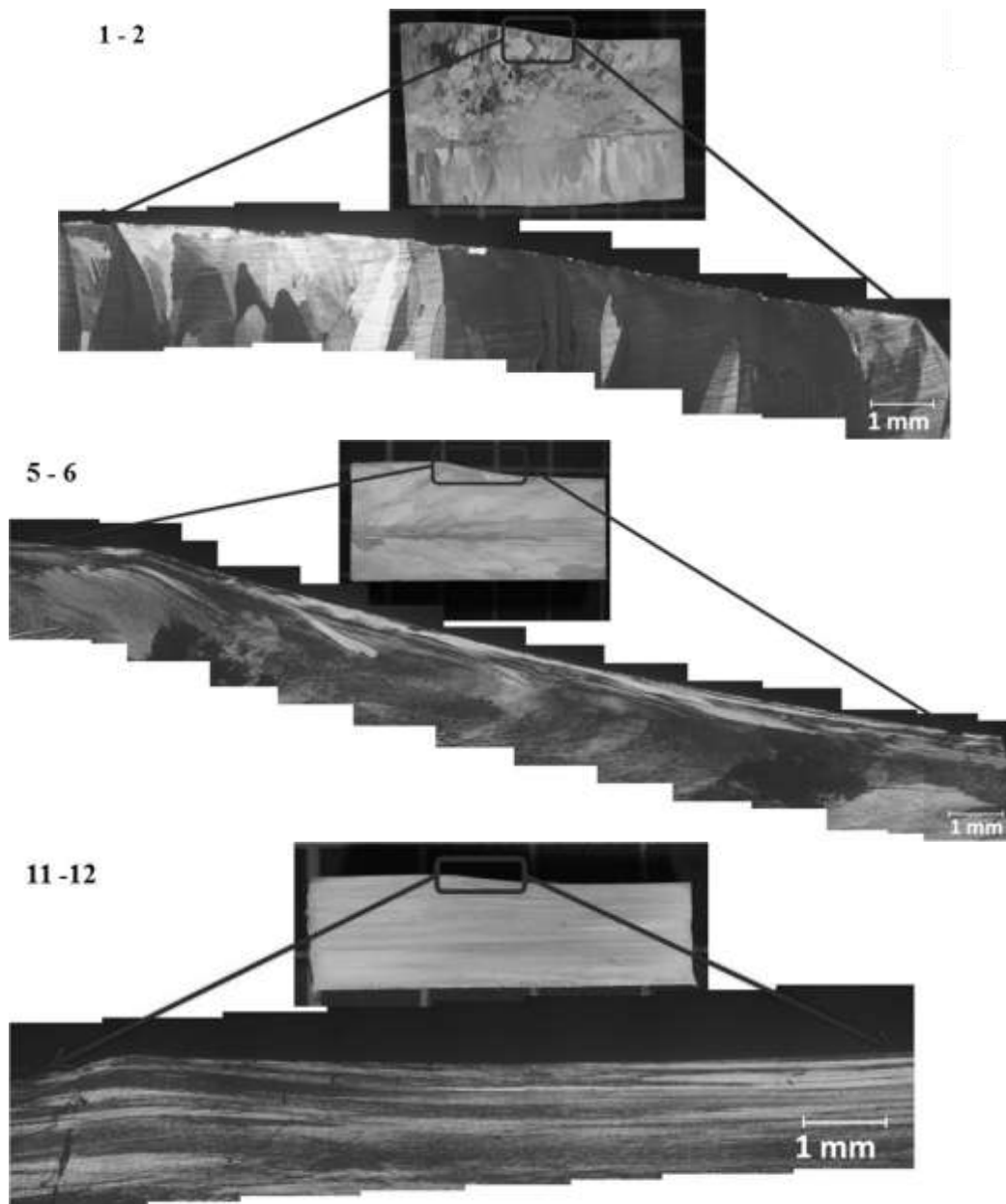
**Figure 4.** The grain structure of the cross-sectional samples after the 1<sup>st</sup>, 5<sup>th</sup> and 12<sup>th</sup> technological steps by macro and Barker etching

As shown in longitudinal section series of Fig. 5, moving from the initial (as-cast) state towards to the last rolling step, the grain structure changes. It is confirmed that despite grain refinement was applied, initially a columnar structure was formed. In the middle of the columnar structure, a nearly equiaxed area (NEA) is visible which is parallel with the longitudinal direction of the strand and is surrounded by perpendicular columnar grains. During rolling, the grains in the NEA and the columnar grains are elongated and rotated into the rolling direction, creating a fiber-like grain structure. After the 7<sup>th</sup> - 8<sup>th</sup> technology steps, the two areas cannot be separated any longer and etching the end of the technology the fiber structure is becoming finer.



**Figure 5.** The grain structure of the longitudinal-sectional samples by macro etching in all technology steps

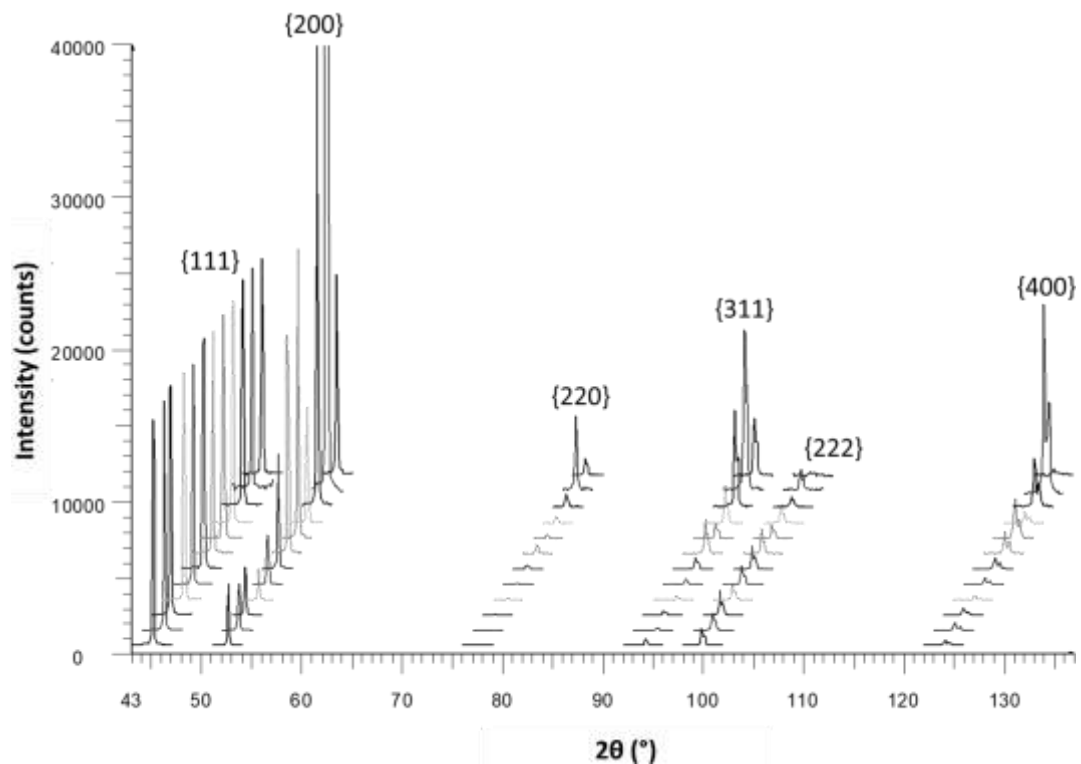
In the longitudinal-sectional sample series the effect of rolling is the most evident at the reduction zones (transition zones) Because of that, Barker etching was performed at these zones Fig. 6 shows the results of the Barker etching in the areas of the 1<sup>st</sup> – 2<sup>nd</sup>, 5<sup>th</sup> – 6<sup>th</sup> and 11<sup>th</sup> – 12<sup>th</sup> transitional zones. The above mentioned same phenomena - the initial columnar structure turns into a fiber structure - can be observed as the technology proceeds. Furthermore, due to the Barker etching, the change in the orientation of the grains can be seen.



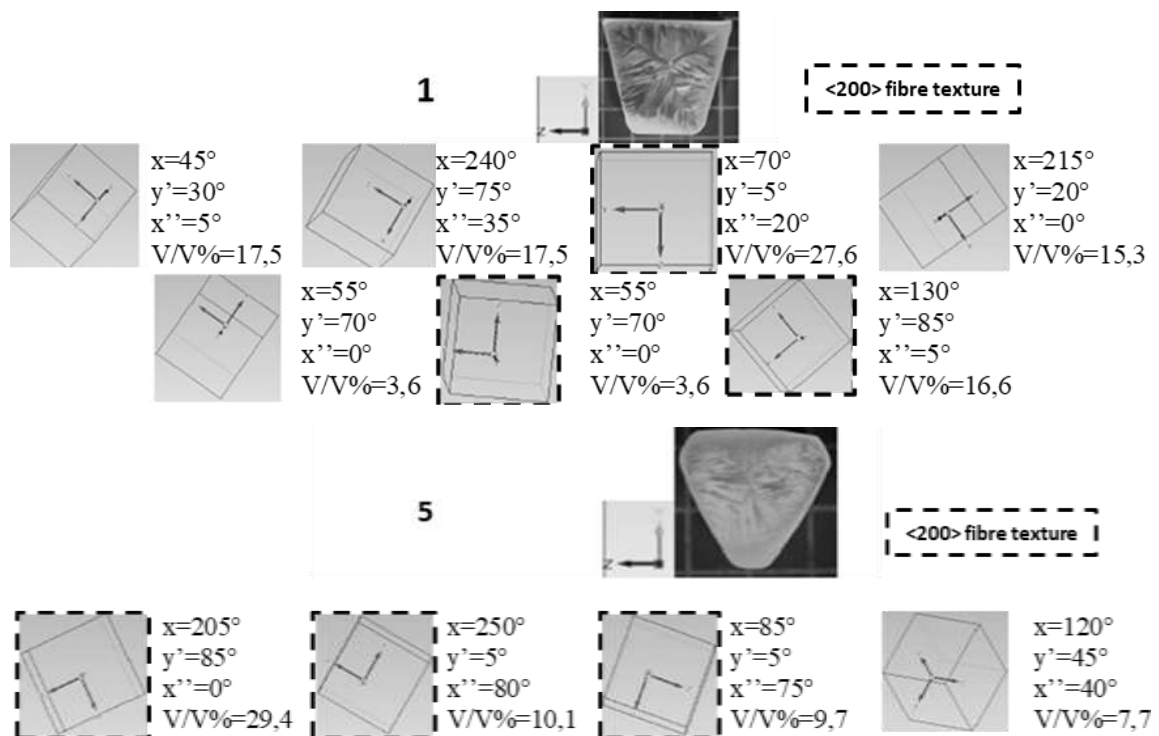
**Figure 6.** The grain structure of the longitudinal-sectional samples between the 1<sup>st</sup> – 2<sup>nd</sup>, 5<sup>th</sup> – 6<sup>th</sup> and 11<sup>th</sup> – 12<sup>th</sup> transitional zones

The full XRD spectra (interference functions) of the circular cross-sectional samples are shown in the Fig. 7. To eliminate the decreasing intensity of the peaks resulting from the decreasing examined volume, the intensity values of the peaks on each XRD spectra have been normalized to the  $\{111\}$  peak of the 12<sup>th</sup> sample. It is shown in Fig. 7 that after the initial technological step, the casting, a strong  $\{200\}$  (equivalent to  $\{100\}$ ) texture developed. As the technology proceeds, a  $\{111\}$  texture is formed. In the final step, a strong  $\{111\}$  texture rules besides the initial  $\{200\}$  texture.

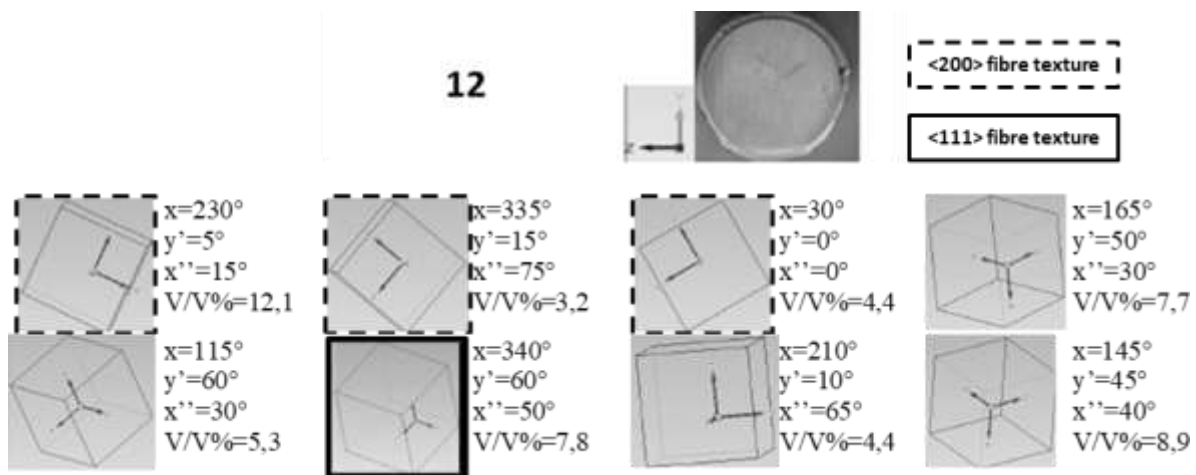
After transforming the calculated ODF into the Properzi coordinate system, the favored orientations of the unit cells and their volume fractions were calculated which presented in Fig. 8. The favored unit cell orientations are in a good agreement with the results obtained from the interference functions.



**Figure 7.** The full XRD spectra of the cross-sectional samples after each technological step







**Figure 8.** The favored orientations of the unit cells after the 1<sup>st</sup>, 5<sup>th</sup> and 12<sup>th</sup> technological steps

#### 4. Summary

Based on the results of grain structure examinations, it can be stated that a strong columnar structure formed in the as-cast state despite of the applied grain refinement. The columnar structure turned into a fibre structure due to the rolling steps and as the technology proceeded, the fibre structure became finer.

Due to the formed columnar structure, a strong {200} texture developed after solidification, which remained in the material as the technology proceeded, although a strong {111} fibre texture developed due to the rolling. After the final step, besides the forming {111} fibre texture, the initially developed {200} fibre texture still can be observed.

It can be stated that the initially developed grain structure and crystallographic texture have a major influence on the final texture of the semi- product, thus, the properties of the semi- product.

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#### References

- [1] U.F. Kocks, C.N. Tomé, H.R. Wenk, Texture and Anisotropy, Cambridge University Press, 1998
- [2] Olaf Engler, Valerie Randle, Introduction to texture analysis, Second Edition, 2010
- [3] Satyam Suwas, Ranjit Kumar Ray, Crystallographic Texture of Materials, 2014
- [4] ASTM E81: 1977 (1982) Preparing Quantitative Pole Figures of Metals
- [5] Hlavács Adrienn, A mikroszerkezet változása Properzi öntvehengerlés során, Diploma thesis, Miskolc, Hungary 2014 (in Hungarian)
- [6] Dr. Bárczy Pál, Dr. Fusch Erik, Metallográfia I., Tankönyvkiadó, 1981 (in Hungarian)