

# Industrial application of a quick, non-destructive anisotropy characterisation method

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**Abstract.** Centerless X-ray diffractometers are specially designed for non-destructive residual stress measurement. The residual stress, and the crystalline anisotropy are induced by elastic deformation and are stored within the crystal structure. The similarity between residual stress and pole figure measurement led to the idea of developing the texture test method for centreless diffractometers. After the determination of identical lattice plane series distribution that can be measured with centreless and conventional diffractometers, advanced anisotropy measurements techniques were invented. In some cases, to characterize the sample from the aspect of crystalline anisotropy it is enough to make measurements in the specific directions (e.g.  $\chi$  cuts in the rolling, cross and diagonal direction). With our centreless X-ray diffractometer, we managed to measure a  $\chi$  cuts in the rolling direction under two minutes without sample cutting. The implementation of this non-destructive technique into the production line can be possible, consequently monitoring of the texture evolution even on industrial-sized aluminium coils. In this paper we compare the conventional X-ray diffraction measuring technique with our developed method on the example of a cold rolled aluminium sheets at different reductions.

## 1. Introduction

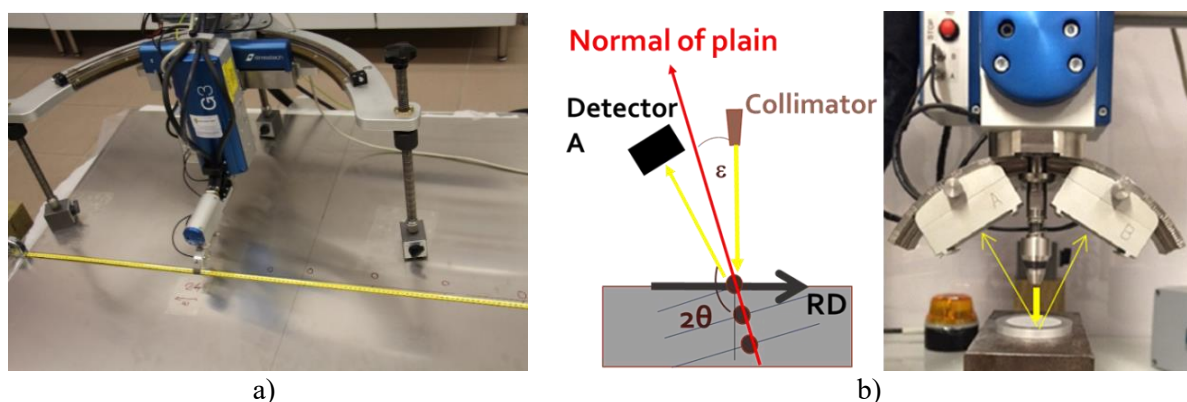
The industrial significance of anisotropy is highly important in metal forming and shaping processes, for example, during the deep drawing, drawability. During this process the earring indicates the homogeneity of the formability. Of course, small earring and homogenous deformation is favoured, which can be achieved by a texture-free structure [1,2,3]. The pure deformation or recrystallization (annealing) texture cause large earring, which can be reduced by an appropriate combination of them. From the casting there are several reasons that influence the final texture after the last technological step in the case of aluminium alloys [4]. Obviously, the effect of annealing (rate of the last cold forming, annealing time, temperature) conditions are crucial, but the formation of anisotropic texture is also influenced by the presence of secondary phases formed during the casting or homogenisation. This is the so-called PSN (Particle Stimulated Nucleation) effect, when the number of dislocations increase in the surroundings of the rigid, not formable particles thus the stress field of these dislocations helps the nucleation of the recrystallization and the forming of isotropic microstructure [4,5,6]. This process highly depends on the size, shape and distributions of the precipitations determined by compositions, casting, preheating prior to hot forming and the conditions of homogenization. Due to this, it is possible to observe different earring rates at the start, middle, at the end and even in cross direction of the rolled aluminium coil. Therefore, very often, the edges of the coil are trimmed to separate the sections have



different drawability. There is an industrially used method to characterise the drawability, called cup drawing. The results of the tests show the cause of earring (deformation or recrystallization), however, such results cannot give an answer with details. Furthermore, it is a destructive measuring technique. Fortunately, the X-ray diffraction can provide detailed information on texture. The only problem with conventional X-ray diffraction technique and the characterization by pole figure or ODF (Orientation Distribution Function) is that the measuring time is too long, and the sample size is limited which requires the need for sample cutting and preparation. To characterize the sample from the aspect of anisotropy, it is enough to make measurements in the preferred directions (e.g.  $\chi$  cuts in the rolling, cross and diagonal direction). With our centreless X-ray diffractometer (Stresstech G3R), we managed to measure a  $\chi$  cuts in the rolling direction under two minutes without sample cutting (Figure 1a). These centreless X-ray diffractometers are originally produced to measure residual stress and retained austenite volume fraction non-destructively and quickly even in industrial environment [7]. The implementation of this non-destructive technique into the production line can be possible, thus, monitoring the texture evolution even on industrial-sized aluminium coils. In this paper we compare the conventional X-ray diffraction measuring technique with our developed method on the example of a cold rolled aluminium sheets.

## 2. Experimental

The measurements were carried out on 3103 type aluminium cold rolled sheets that were rolled with small steps. The measurements were performed after each reduction at the following sample thickness: 5.74 mm; 5.36 mm; 4.97 mm; 4.49 mm; 4.01 mm; 3.52 mm; 3.04 mm; 2.54 mm; 2.05 mm; 1.58 mm; 1.16 mm; 0.85 mm; 0.68 mm and 0.45 mm. The  $\chi$  cuts were measured on the  $\{222\}$  plane series. For the conventional pole-figure measurements, a Bruker D8 Advance diffractometer was used equipped with Eulerian cradle. The details of the conventional measurement were:  $\text{CoK}_\alpha$  radiation, 40 kV acceleration voltage, 40 mA tube current. With this technique the  $\{222\}$  pole figures were measured, and the  $\chi$  cuts in the rolling direction were selected from these pole figures. The non-destructive measurements were performed with the Stresstech G3R device in OMEGA mode,  $\text{CrK}_\alpha$  radiation was used with 3 mm collimator and with 2 second exposure time. Figure 1 shows the measuring setup and the beam path.

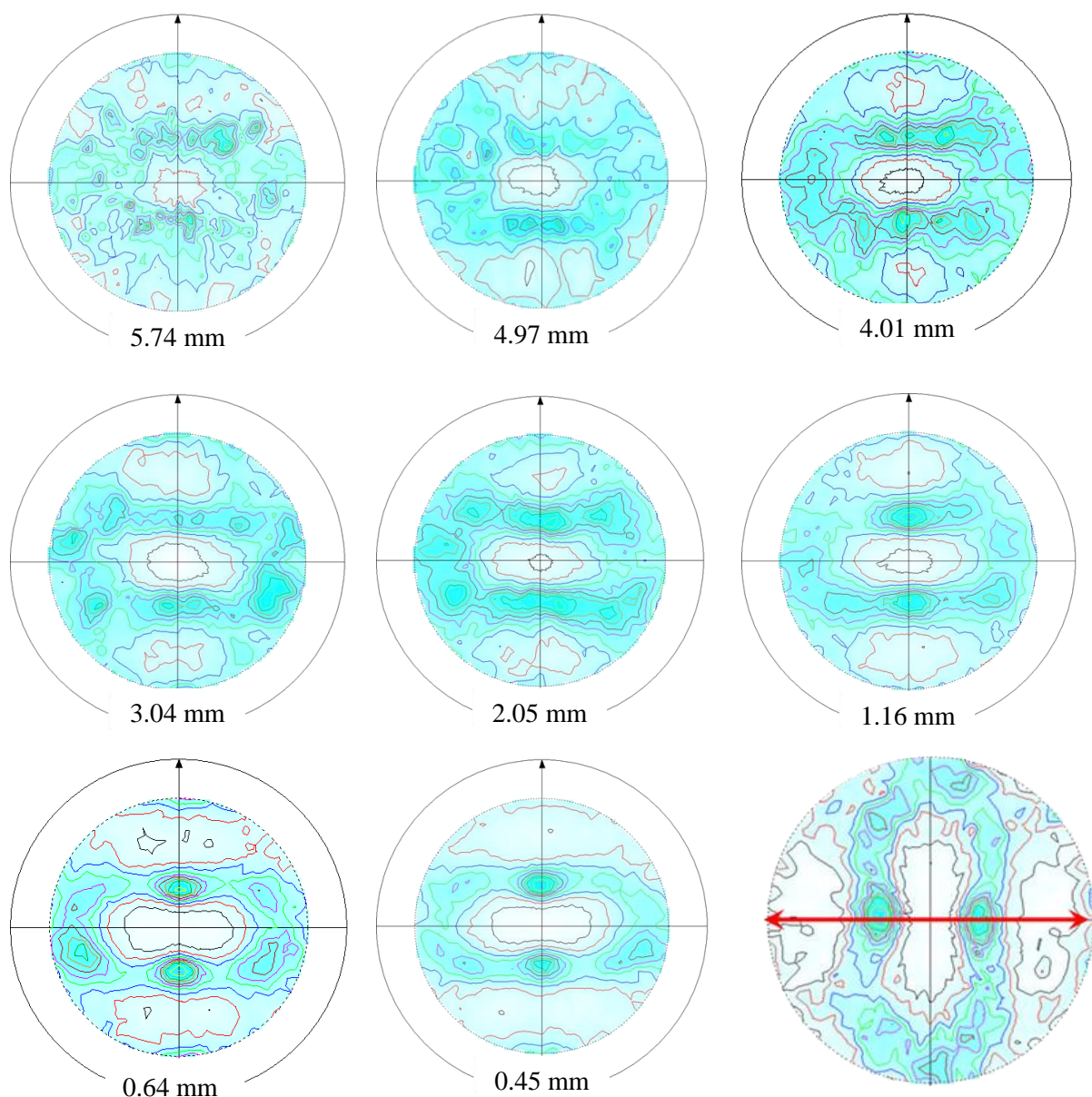


**Figure 1.** Non-destructive measuring with a Stresstech G3R device a) measuring of the aluminium sheet b) beam pass in OMEGA mode (schematic and photo - with A detector, Rd-rolling direction)

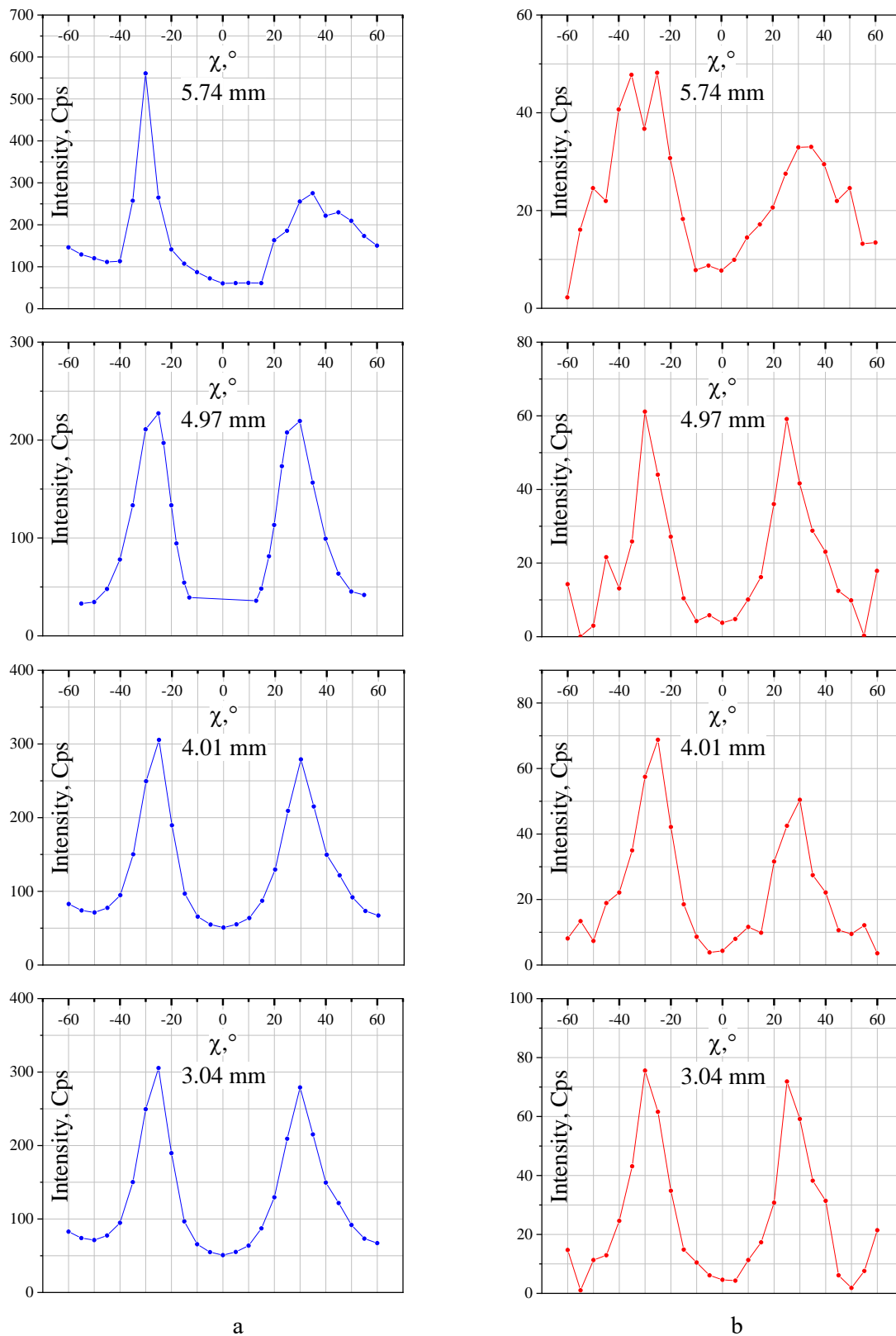
## 3. Results and discussions

Figure 2. shows the measured  $\{222\}$  pole figures on the specimens obtained with the Bruker D8 Advance. It is observable, how the rolling deformation texture builds up with higher reduction, the characteristic of the deformation texture getting stronger and stronger as it was expected. The last pole

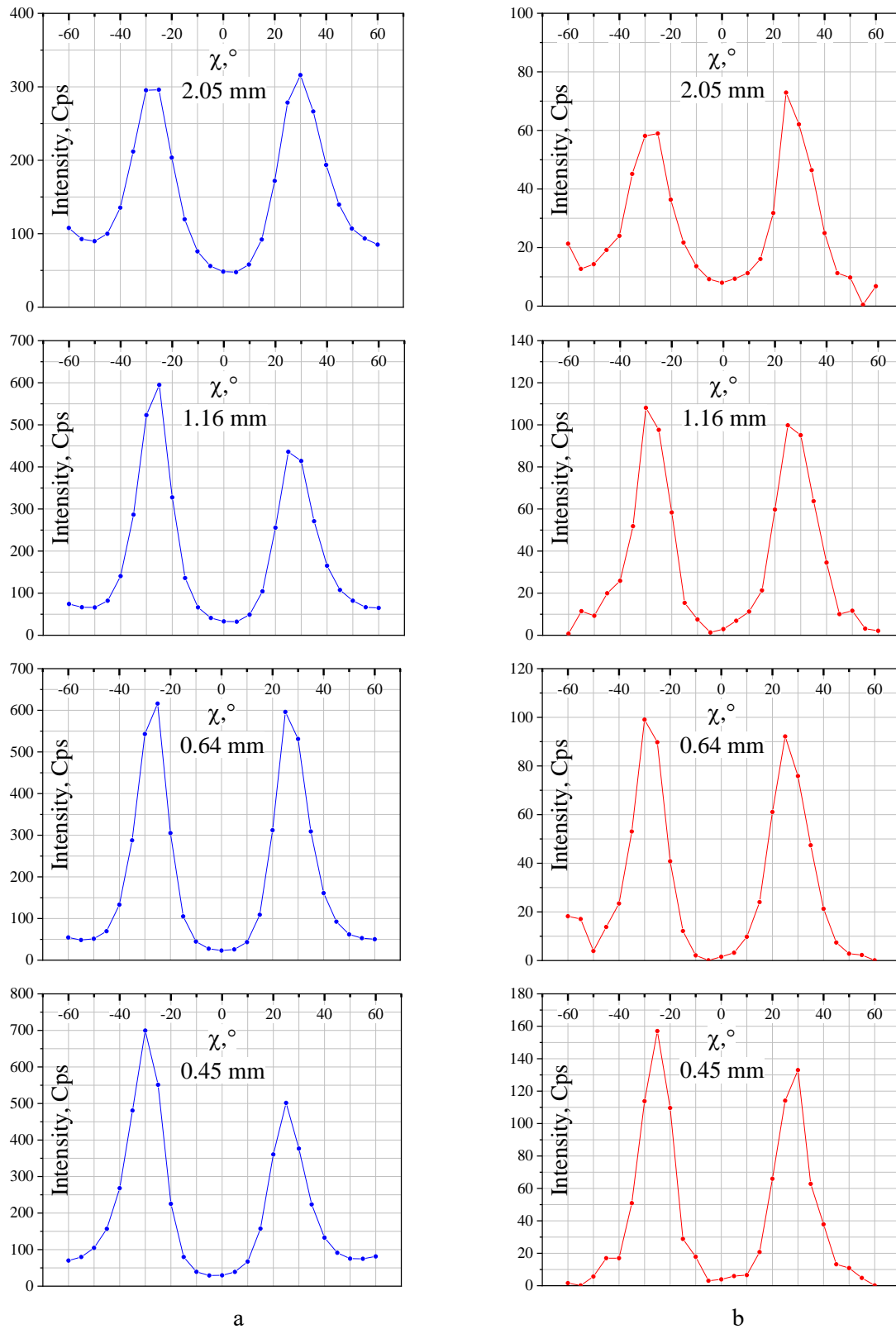
figure is rotated with 90° degree, and the red arrow shows the interpretation of the  $\chi$  cuts in the rolling direction. Figures 3-4 represent the  $\chi$  cuts, the blue cuts show the non-destructive technique measured directly with the Stresstech G3R, and the red cuts measured with the Bruker D8 Advance, calculated from the pole figures. The reason of the asymmetric characteristic of the individual curves is that asymmetric tribological conditions were built up in some cases. Comparing the conventional and the non-destructive methods, the same tendency is observable. Even the above-mentioned asymmetry is revealed and fits well for both equipment, for example, at the 5.75 or 0.45 mm thickness. As the specimens suffered higher and higher deformation, the results measured with the different techniques fit better and better to each other, since the texture is increased in the examined volume, and the texture gradient getting smaller. This agrees with the results of the pole figures. On the y axes the absolute intensity scales are not the same because they were measured by different devices and parameters, but the tendency is absolute convincing.



**Figure 2.** Rolled 3103 type aluminium {222} pole figures after different reduction (5.74 mm-0.45 mm), red arrow shows the interpretation of the  $\chi$  cuts in the rolling direction



**Figure 3.**  $\chi$  cuts of aluminium {222} pole figures after different reductions (from 5.74 mm to 3.04 mm) in the rolling direction a) measured by centreless X-ray diffractometer (Figure 2) b) from the conventionally determined pole figures



**Figure 4.**  $\chi$  cuts of Aluminium {222} pole figures after different reduction (from 2.05 mm to 0.45 mm) in the rolling direction a) measured by centreless X-ray diffractometer (Figure 2) b) from the conventionally determined pole figures



#### 4. Conclusions

The formation of anisotropy plays very important role the metal forming industry, and there are several methods to characterize it. Unfortunately, most of these methods are destructive, sample-cutting, and preparation always needed. In this paper, a unique non-destructive measurement technique was shown based on X-ray diffraction and compared with the conventional X-ray diffraction technique. The advantages of this method are obviously its non-destructive nature and the very short measuring time (~2 min.) what create the justification for implementation into the industry. Validation measurements were performed with a Stresstech G3R centreless X-ray diffractometer and a conventional, Bruker D8 Advance X-ray diffractometer equipped with an Eulerian cradle. The characterization of anisotropy by the pole figure is a well-established method, but it takes a long time. In some cases, especially industrial environment, it is enough to perform measurements in the specific directions (e.g.  $\chi$  cuts in the rolling, cross and diagonal direction). With our centreless X-ray diffractometer, we managed to measure a  $\chi$  cuts in the rolling direction under two minutes without sample cutting. The validation measurements were performed on a 3103 type aluminium alloy through 8 steps rolling. The results determined by the two methods were identical, even the effect of asymmetric tribological conditions was able to determine.

#### Acknowledgments

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#### References

- [1] Engler O and Randle V 2010 *Introduction to texture analysis* (CRC Press).
- [2] Suwas S and Ray K R 2014 *Crystallographic Texture of Materials* (London: Springer-Verlag)
- [3] Engler O 2012 *Materials Science and Engineering A* **538** pp 69-80
- [4] Kocks F, Tomé C and Wenk H 1998 *Texture and Anisotropy- Preferred Orientations in Polycrystals and their Effect on Materials Properties* (Cambridge: Cambridge University)
- [5] Rollet A, Humphreys F, Hatherly M 2012 *Recrystallization and Related Annealing Phenomena* (Oxford:Elsevier) pp 256-274
- [6] Vatne H, Engler O and Nes E 1997 *Materials Science and Technology* **13** pp 93-102
- [7] ed Totten G, Howes M, and Inoue T 2008 *Handbook of Residual Stress and Deformation of Steel* (Materials Park: ASM International)