Microstructural study of duplex stainless steel after turning using EBSD and X-ray diffraction analysis

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Abstract. The aim of this research is a microstructural study of duplex stainless steel after turning. The electron backscatter diffraction and X-ray diffraction analyses were applied to analyse the surface and subsurface regions. The gradients of Kernel Average Misorientation as a function of flank wear were investigated. It was found that the concentration of deformation depends on the subsurface depth, individual phases, and flank wear. The similar behaviour but with a different inclination of grain size (determined by electron backscatter diffraction) and crystallite size (determined by X-ray diffraction) depending on flank wear was observed.

1. Introduction
Duplex steels are dual-phase steels contain austenitic-ferritic microstructure with a similar proportion of both phases. Due to the combination of properties of both phases and mainly two-phase microstructure, duplex steels exhibit high corrosion and wear resistance in many environments, where high-alloyed single-phase steels are usually used [1]. The mechanical properties of austenite and ferrite are generally different; therefore, the properties of duplex steel strongly depend on microstructure (e.g. grain size, texture) and others (phase content, macro- and microstresses, chemical composition, etc. [2, 3]).

Our team [4] verified that major deformation mechanisms are the same as in the single-phase steels. Nevertheless, due to inter-phase interaction, their behaviour during deformation can be different in comparison with single-phase steels [5]. Therefore, the statement that austenite and ferrite phases are generally plastic or elastic, is not generally valid.

According to [2], the strain-hardening rate of austenite is higher than for ferrite. However, it is lower in comparison with single-phase austenitic stainless steel with the same chemical composition due to the very low stacking fault energy. The explanation of this behaviour is inter-phase interaction which redistributes the plastic deformation into both phases. The strain-hardening of austenite is very high at low deformations, but from certain deformation, it increasingly concentrates within the ferrite phase, which has a larger number of active slip systems and a considerably higher stacking fault energy. In other words, the austenite phase becomes harder than ferrite phase. Wroński et al. [6] and Choi et al. [7] investigated the plastic deformation in duplex steel by electron backscatter diffraction. The samples...
studying after tensile deformation brought the conclusion that the Kernel Average Misorientation (geometrically necessary dislocations — their density) increases faster in the austenite phase than in the ferrite phase during deformation.

During plastic deformation, the high dislocation density is generated and form low- and high-angle boundary. According to the generally known definition, the high-angle boundaries delimit grains and low-angle boundaries so-called sub-grains (in limit crystallites). Our team [3] besides other things found that the crystallite size of the plastically deformed surface layer is nearly unaffected with flank wear. Afterwards, the gradient of crystallite size decreases with flank wear due to higher dislocation density in deeper regions. Our previous study of duplex steels, Capek et al. [8], shows the influence of crystallite size depth profiles concerning inserts rake angle. We found that the gradient of crystallite size was similar for all rake angles but, the higher crystallite size was evaluated for positive rake angles. Moreover, microhardness depth profiles were steeper for positive rake angles. All used inserts were new, i.e. sharp; thus, the influence of wearing tool should be the next step.

Therefore, this study focuses on the study of the microstructure of duplex steel after turning with defined conditions (different tool wear) using the electron backscatter diffraction and X-ray diffraction analyses. The investigations of Kernel Average Misorientation, grain and crystallite size are pointed out.

2. Material and experimental methods
Experiments were carried out on the coarse-grained duplex steel SAF 2205 (EN 1.4470) with balanced ferrite and austenite content. Chemical composition is shown in Table 1.

| Table 1. Chemical composition of the duplex steel 1.4470 (wt. %) [2]. |
|-------------------|---|---|---|---|---|---|---|---|
| Fe                | C  | Mn | Mo | Cr | Ni | S  | P  | Si | N  |
| bal.             | 0.015 | 1  | 3  | 22 | 5.5 | 0.012 | 0.017 | 0.5  | 0.16 |

The specimens were face dry turned. Cutting conditions were as follows: cutting speed 110 m/min⁻¹, cutting depth 1 mm, feed 0.1 mm, tool holder DWLN R 2525 M08 KT 80 and cutting inserts SNMG 120408 of grade P20 with TiN coating and flank wear width (the average width of the worn area on the cutting edge) VB = 0.05, 0.12, 0.25, 0.4, and 0.53 mm.

Deformation induced to the surface of turned samples was investigated by electron backscatter diffraction on the CD/ND cuts¹. The samples were analysed in Auriga Compact SEM equipped with EDAX EBSD camera. The EBSD mapping was performed near the turned surface with the scan size $200 \times 400 \mu m^2$ and the resolution of 0.25 μm. A 10×10 grid surrounding the pixel of interest with threshold 5° was used during each misorientation calculation to obtain Kernel Average Misorientation. Experimental data were processed using the MATLAB toolbox MTEX software [9].

X’Pert PRO MPD diffractometer with cobalt radiation was used to the analyses of CD/TD plane of the samples by X-ray diffraction (XRD). Crystallite size was calculated using the Scherrer formula. The irradiated area was defined by experiment geometry, effective penetration depth of the XRD radiation (approx. 5 μm) and pinhole size (4×0.25 mm²). To analyse the gradients beneath the sample surface, layers of material were gradually removed by electrochemical polishing using the Struers LectroPol-5 device.

¹ CD, TD, ND denote cutting, transversal, normal direction, respectively.
3. Results and discussions
For a microstructural study of grain size, crystallite size and Kernel Average Misorientation (KAM), the X-ray diffraction (XRD) and electron backscatter diffraction (EBSD) were used.

3.1. Kernel Average Misorientation
The Kernel Average Misorientation is calculated as the average misorientation between each pixel and its 3rd nearest neighbours. The gradient of KAM was investigated from the point of view of the difference between both phases and the flank wear VB. To obtain one KAM value from each depth, the values from the 4 μm subsurface layer were averaged. The average KAM distributions of both phases in comparison with flank wear are depicted in Figures 1 and 2. Due to this local misorientation from the presence of geometrically necessary dislocations, it can be said that with higher plastic deformation (higher VB), more dislocations are created near the surface and therefore, higher KAM values are observed.

As a result of the strong plastic deformation and surface roughness, the surface regions show a higher proportion of non-indexed points, see Figure 1, nevertheless, high KAM values could be expected. The results also show that high KAM value was detected around the phase boundaries.

The KAM values in both phases in relation to the flank wear VB are depicted in Figure 3. For low plastic deformation (VB = 0.05 mm), the austenite is due to stacking fault energy more plastic, i.e. higher KAM is for the ferrite. However, the strain-hardening is higher in the austenite than in the ferrite at low deformations and, therefore, the KAM values are higher for austenite phase for higher VB (from approx. 0.25 mm). These results are in agreement with the authors previous researches of texture behaviour [4] and residual stresses of duplex steels [8]. In these articles as well as in the literature [6], the influence of different strain hardening was observed.

Figure 1. The sections of the KAM maps for each VB.
3.2. Grain size vs. crystallite size

The grain size ($GS$) was evaluated from EBSD data as an average of distances between high-angle boundaries (above 10°) in the CD direction\(^2\). Due to high data dispersion (follows from coarse-grained microstructure) and for better clarity, the $GS$ was fitted by a sigmoid function. The average $GS$ of bulk material is around 15 μm, see Figure 4. The crystallite size ($D$) was evaluated from the X-ray diffraction pattern using Rietveld refinement averaged from both phases in the CD direction. The maximum value of the $D$ is 500 nm given by used MStruct program [11] and the presented error follow from Rietveld analysis.

For both cases ($GS$ and $D$), the samples with higher $VB$ reach the maximum $GS$ in the greater depth. Figures 4 depict the comparison of the gradient of $GS$ and $D$ depending on $VB$, in other words, show the difference of development of high- and low-angle boundaries with subsurface depth. Because the values of $D$ are dependent on the rate of plastic deformation (more precisely dislocation density), $D$ is affected by turning more than $GS$, see Figure 5. With higher

\(^2\) See [10] function grains.boundary.intersect.
VB, this difference between GS and D is bigger. For \( VB = 0.53 \) mm, D reach the maximum even above 350 \( \mu \)m of subsurface depth. The explanation of this phenomenon resides in the forming of shear bands. Due to high dislocation density in these bands, much finer crystallites are generated in contrast with grains [12]. Because the low-angle boundaries define both KAM and D, the comparison of the KAM values with reverse D is comparable, see Figure 6.

Figure 5. Comparison of the gradient of grain size (GS) and crystallite size (D) depending on VB.

Figure 6. Comparison of the gradient of KAM and reverse crystallite size (1/D) with respect to VB.

4. Conclusions
The following results were obtained:

- The steep gradient of KAM was found with respect to flank wear. Affected subsurface depth of turned material gradually grows with higher VB.
- The different strain hardening rate was found for each phase.
- The steep gradients of grain and crystallite size were found.
- Flank wear influence the affected subsurface depth with higher dislocation density.
- The gradients of KAM values and reverse D have comparable trends.

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