

Article

Investigating the Influence of Process Parameters on the Mechanical Properties of Extruded Aluminum Tubes by Cyclic Indentation Tests

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Abstract: Given the complex process condition, extruded aluminum (Al) alloy tubes show locally pronounced differences in microstructure and mechanical properties, which can be influenced by subsequent heat treatment. In the present study, cyclic indentation tests (CITs) were conducted on extruded Al alloy EN AW-6082 to locally determine hardness and cyclic hardening potential, which was complemented with light optical microscopy. To analyze the influence of extrusion process and subsequent heat treatment, the EN AW-6082 tubes investigated were manufactured with extrusion ratios Ψ of 13:1 and 22:1, both in as-extruded and T6 heat-treated conditions. The results obtained for the as-extruded state showed significant differences of the local mechanical properties and demonstrated that an increased Ψ leads to higher hardness, caused by more pronounced plastic deformation during the manufacturing process. Moreover, an increase of hardness and cyclic hardening potential was observed after a T6 heat treatment, which also reduced the difference in hardness between the different extrusion ratios. Additionally, the pronounced local differences in hardness and cyclic hardening potential correlated with the local microstructure. The results demonstrated that CITs enable the analysis of local mechanical properties of extruded EN AW-6082 profiles, resulting from different extrusions ratios as well as subsequent heat treatment.

Keywords: cyclic indentation test; cyclic hardening potential; extruded Al tubes; PhyBaL_{CHT}



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1. Introduction

The ongoing trend in the transportation industry to save weight by the downsizing of components also increases the material requirements. Besides the corrosion resistance and recyclability, the reliability of lightweight components under service conditions has to be considered [1]. Therefore, a sound knowledge of the mechanical and especially cyclic properties of lightweight materials is a prerequisite and, thus, of great industrial interest. This is underlined by broad applications of lightweight components made of aluminum (Al) alloys in the automotive and aerospace industry. In this context, thin-walled structures are used to improve crash resistance and safety while not increasing overall weight [2]. The potential of such structures for absorption of mechanical energy depends primarily on their shape, with cylindrical tubes absorbing the most energy per unit mass [3].

In addition to the shape, several mechanisms, i.e., grain refinement, strain hardening, solid solution strengthening, and precipitation hardening, increase the strength of Al alloy tubes, depending on the production process used, e.g., hot extrusion. Besides the alloy used, the temperatures and amount of plastic deformation applied during manufacturing have an impact on the mechanical properties of Al alloy tube and thus, have to be considered [4]. Work hardening by cold plastic deformation increases the strength of the material,

which can be attributed mainly to an increase in dislocation density [5,6]. This leads to a decreased potential for energy absorption because of the lower ductility after cold plastic deformation [7–9].

Wrought Al-Mg-Si alloys are commonly used for production of lightweight tubes and the final mechanical properties can be tailored by suitable heat treatments. Therefore, it has to be considered that the high amount of plastic deformation occurring in extrusion processes accelerates the kinetics during precipitation hardening of Al tubes. Consequently, the aging temperatures or the aging times have to be decreased, depending on the actual process-induced material condition [10].

In addition to the mentioned mechanisms for strength increase, two mechanisms are known that result in a decreased strength in these alloys: recrystallization and recovery. During recrystallization, point defects and dislocations created by deformation are eliminated by the formation of new grains, leading to a reduction of the internally stored energy. The deformed microstructure is replaced by a recrystallized microstructure that exhibits similar mechanical properties as the undeformed material [5,11]. Recovery mostly influences dislocations, which can be rearranged or annihilated. It should be noted that a distinction is made between dynamic recrystallization and recovery as well as static recovery, depending on the process kinetics. The dynamic processes occur during plastic deformation of the material at elevated temperatures, such as hot extrusion, and lead to a modification of microstructure, whereby only dynamic recrystallization leads to changes of the grain structure. In contrast, static recovery results from thermally controlled processes, i.e., annealing performed after the forming process, which does not affect the grain structure [5,11].

As these microstructural processes can, due to highly inhomogeneous amounts of plastic deformation, be locally different in extruded Al tubes, small-scale testing methods are required for analyzing the local mechanical properties. For this, indentation testing can be used, which enables the local characterization of a material's monotonic mechanical properties, i.e., hardness or elastic modulus [12–14]. By performing cyclic indentation tests (CITs), the cyclic properties of a material, e.g., the cyclic deformation behavior [15,16] or fatigue crack propagation [17], can also be analyzed. In addition, CITs can be used to determine the cyclic hardening potential of metallic materials, which correlates well with the amount of cyclic hardening obtained in uniaxial fatigue tests, as shown for the steel 18CrNiMo7-6 [15] and for two Cu-alloyed steels alloyed with carbon contents of 0.005 wt.% and 0.2 wt.%, respectively [18]. Besides the correlation with cyclic hardening potential, it was shown in [18] that considering the cyclic hardening potential enables a better assessment of fatigue strength compared to taking into account only hardness. Furthermore, it was demonstrated for differently heat-treated 42CrMo4 that the cyclic deformation behavior detected in CITs is similar to the cyclic deformation curves obtained in uniaxial fatigue tests at cyclic compressive stresses [19].

In many earlier investigations, the mechanical properties determined by indentation testing were linked to microstructural mechanisms. As shown by Lim et al. [20] for high purity copper and aluminum as well as by Weiler et al. [21] for die-cast magnesium AM60B alloy, the hardness and thus the strength increase with decreasing grain size, which can be explained by the well-known Hall–Petch effect [22,23]. Furthermore, by using CITs, Kramer et al. [15] demonstrated that the cyclic hardening potential of 18CrNiMo7-6 increases with decreasing grain size. Moreover, Schwich et al. [24] showed an interrelation of the cyclic deformation behavior determined in CITs and the morphology of nanoscaled Cu precipitates in a Cu-alloyed steel with 0.005 wt.% C.

By decreasing the indentation force, the cyclic properties of a material can be analyzed more locally [19], which was successfully used in [25] to characterize the cyclic properties of various surface morphologies of high-Mn HSD[®]600 TWIP steel, resulting from different machining processes. Furthermore, Blinn et al. [26] showed that changes in cyclic hardening potential in the vicinity of a fatigue crack can be determined by means of CITs with small

indentation forces. Consequently, CITs are well suited to locally characterize the cyclic properties of metallic materials.

Therefore, in the presented study CITs were used to analyze the influence of different extrusion ratios and, thus, different amounts of plastic deformation on the mechanical properties of extruded Al tubes made of EN AW-6082. Moreover, light optical microscopy was performed to correlate the mechanical properties observed with changes of the microstructure within the Al tubes. Additionally, the as-extruded state was compared to a T6 heat-treated state to investigate the impact of this additional heat treatment on the local mechanical properties.

2. Materials and Methods

The profiles made of the aluminum alloy EN AW-6082 were produced by direct extrusion carried out with a 2.5 MN extrusion press (Müller Engineering, Todtenweis, Germany). For the direct extrusion process, the temperatures of the billet, the die, and the recipient were set to 450 °C and a relatively low ram speed of 2 mm/s was used. Both the billet and the die were preheated to 450 °C outside the extrusion press by means of a forced-air chamber furnace and subsequently inserted into the recipient and the tool holder. The recipient had integrated heating cartridges in order to maintain a constant temperature, which was regulated by the control system (SIMATIC S7-1200, Siemens AG, Munich, Germany) of the extrusion machine. Moreover, the temperature inside the tool was monitored by means of a thermocouple positioned close to the bearing surface. Two billets, each having an outer diameter of 58.4 mm, which was equivalent to the inner diameter of the recipient, and an axial bore hole adapted to the diameter of the respective mandrel (Ø12 mm and Ø16 mm), were used to produce two types of tubes with different wall thicknesses. To reduce friction in the extrusion process, the die and the mandrel were coated with a layer of molybdenum disulfide (MoS_2) as a lubricant. Figure 1 illustrates a schematic of the extrusion process. The EN AW-6082 billet is shown in two shades of blue to illustrate schematically the material flow during direct extrusion in accordance with Ostermann [10].

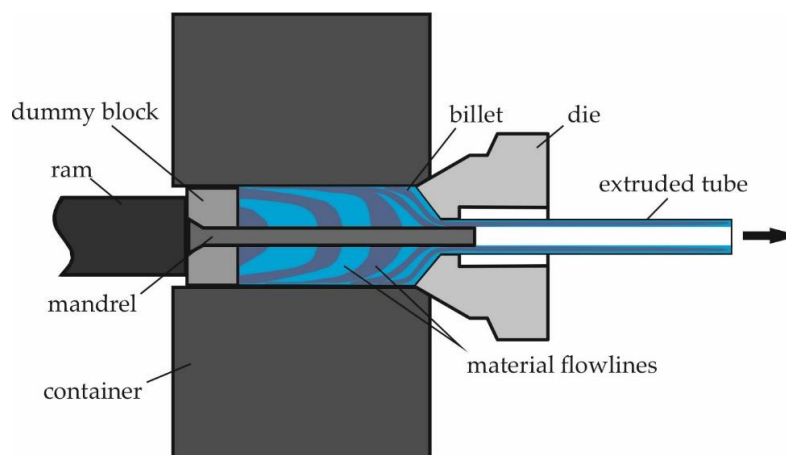


Figure 1. Schematic of the extrusion process of the Al tubes with the estimated material flow based on Ostermann [10].

The extruded profiles had both an outer diameter D_o of 20 mm and differed in internal diameters D_i , being 12 mm and 16 mm, resulting in extrusion ratios Ψ of 13:1 and 22:1, respectively. This resulted in wall thicknesses t of 4 mm and 2 mm, and, thus, the ratio t/D_o of wall thickness to outer diameter was 20% and 10%, respectively.

The exact chemical composition of the EN AW-6082 alloy used was determined by optical emission spectrometry (OES) with a spark spectrometer “SPECTROMAXx” (SPECTRO Analytical Instruments GmbH, Kleve, Germany) and is given in Table 1.

For microstructural analysis as well as indentation testing, cross and longitudinal sections were extracted from the middle part of the extruded profiles using a wet grinder

“Brillant 265” (ATM GmbH, Mammelzen, Germany). The cross sections were taken perpendicular to the longitudinal axis of the profile and the longitudinal sections were extracted parallel to the longitudinal axis along the symmetry line, as schematically shown in Figure 2. The extracted samples were ground and subsequently polished for indentation testing and additionally etched according to Barker for microstructural investigations. For the electrolytic etching according to Barker, 5 mL of tetrafluoroboric acid (HBF₄, 32%, p.a., PANREAC APPLICHEM) were diluted with 200 mL of distilled water. Light optical microscopy of the visualized microstructure was performed with an optical light microscope (“Axioplan 2” from Carl Zeiss AG, Oberkochen, Germany), using a CCD camera (“ProgRes 2008” from Jenoptik AG, Jena, Germany) and polarized light.

Table 1. Chemical composition of the investigated aluminum alloy EN AW-6082 in wt.%.

Element	Si	Mg	Mn	Fe	Cu	Cr	Al
Content	0.82	0.98	0.55	0.27	0.07	0.07	97.10

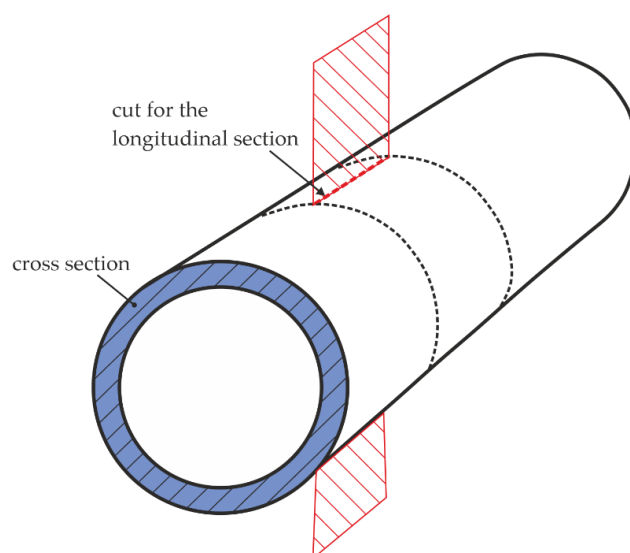


Figure 2. Schematic of the arrangement of the cross sections and longitudinal sections on the extrusion profile.

The samples were analyzed in the extruded condition (as-extruded) as well as after a T6 heat treatment, as suggested by Nowak [4]:

- Solution annealing at 540 °C for 20 min,
- Quenching in water, maximum 2-min holding time until the onset of artificial aging,
- Artificial aging at 180 °C for 4 h.

The mechanical characterization of the investigated conditions was then realized by cyclic indentation tests (CITs), which were performed on a Fischerscope H100c device (Helmuth Fischer GmbH, Sindelfingen, Germany) with a Vickers indenter. These tests were performed on the same sections used for microstructural characterization. In the CITs, a sinusoidal load–time function with a maximum indentation force of $F_{\max} = 1000$ mN and a frequency of $f = 1/12$ Hz was used. As indicated schematically in Figure 3a, the indentations were made at a distance of at least 200 μm from the free surface. Moreover, a minimum distance of 300 μm between each indentation was maintained. In order to characterize the mechanical properties in dependency of the distance to the outer and inner surface, the indentations were placed at different distances d from the outer surface (Figure 3a). To obtain statistically reliable data, mean values and the 90% confidence intervals were determined from at least 40 indentation points per surface distance, whereby 10 cycles were performed during each single CIT.

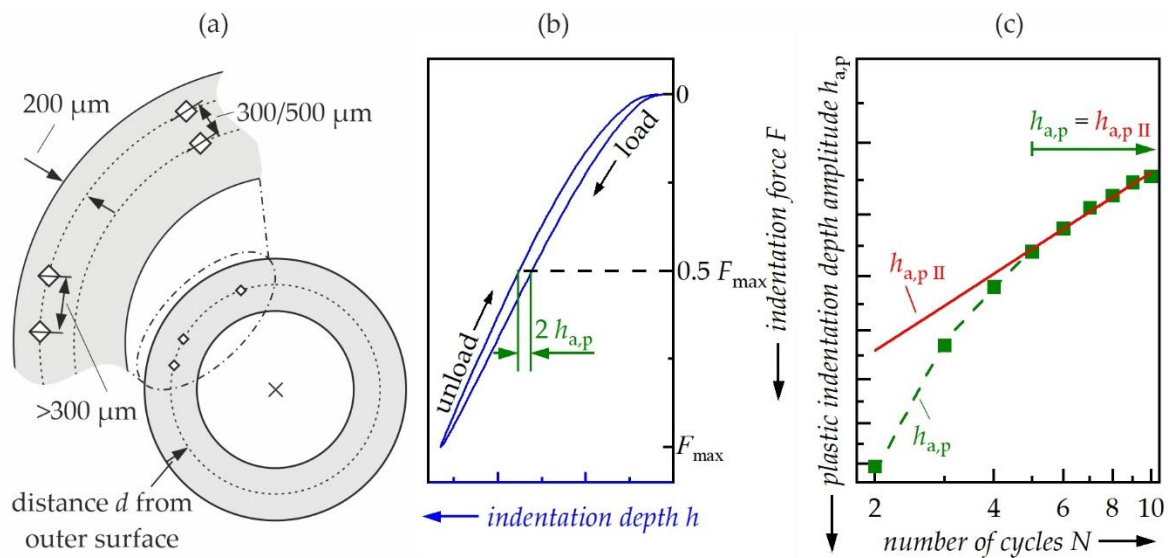


Figure 3. (a) Schematics of a cross section of an extruded Al tube with the positions of indentations and the PhyBaL_{CHT} procedure with (b) hysteresis of indentation force F and indentation depth h including the definition of the plastic indentation depth amplitude $h_{a,p}$ and (c) resulting $h_{a,p}$ – N curve with power function $h_{a,p II}$ [24].

The performed CITs can be evaluated by the PhyBaL_{CHT} approach to characterize the cyclic deformation behavior of the investigated material [15]. For this, indentation force F and indentation depth h are measured continuously during instrumented cyclic indentation. From the second cycle on, an F – h hysteresis was formed in CITs (Figure 3b), which is used for analyzing the cyclic deformation behavior. In analogy to stress–strain hysteresis loops, the half-width of this hysteresis at mean loading, which is indicated as the plastic indentation depth amplitude $h_{a,p}$, is used to characterize the cyclic plastic deformation. The typical evolution of $h_{a,p}$ vs. the number of cycles N is depicted in Figure 3c. Please note that the graph is a schematic and, therefore, no units and specific values are provided. In general, the values of $h_{a,p}$ are in the regime of hundreds to tenths of micrometers (see Figure 4). Figure 3c shows that the slope of the $\log h_{a,p}$ – $\log N$ curve, which represents the cyclic deformation behavior, stabilizes from the fifth cycle on. This indicates that macro-plastic deformation becomes saturated and microplasticity dominates the cyclic deformation behavior in this regime. Hence, the experimentally obtained values of $h_{a,p}$ between the cycles 5–10 can be described by the power function $h_{a,p II}$ [15]:

$$h_{a,p II} = a_{II} \cdot N^{e_{II}} \quad (1)$$

The slope of $\log h_{a,p II}$ vs. $\log N$ is given by the exponent e_{II} , which is called the cyclic hardening exponent_{CHT}, representing the amount of cyclic hardening observed in the CITs. As a higher $|e_{II}|$ is associated with a steeper slope of $h_{a,p II}$ and, thus, more pronounced cyclic hardening, it indicates a higher cyclic hardening potential. In addition, the vertical position of the $h_{a,p}$ – N curves can be associated with the cyclic plasticity of the material, whereby lower $h_{a,p}$ values indicate a lower cyclic plasticity. Please note that, in accordance with [15], the vertical $\log h_{a,p}$ axis (Figure 3c) is shown reversed, i.e., smaller $h_{a,p}$ values are shown further up on the axis. Moreover, based on the indentation depth of the first cycle, Martens hardness HM can be determined. A more detailed description of the PhyBaL_{CHT} approach is given in [15,19].

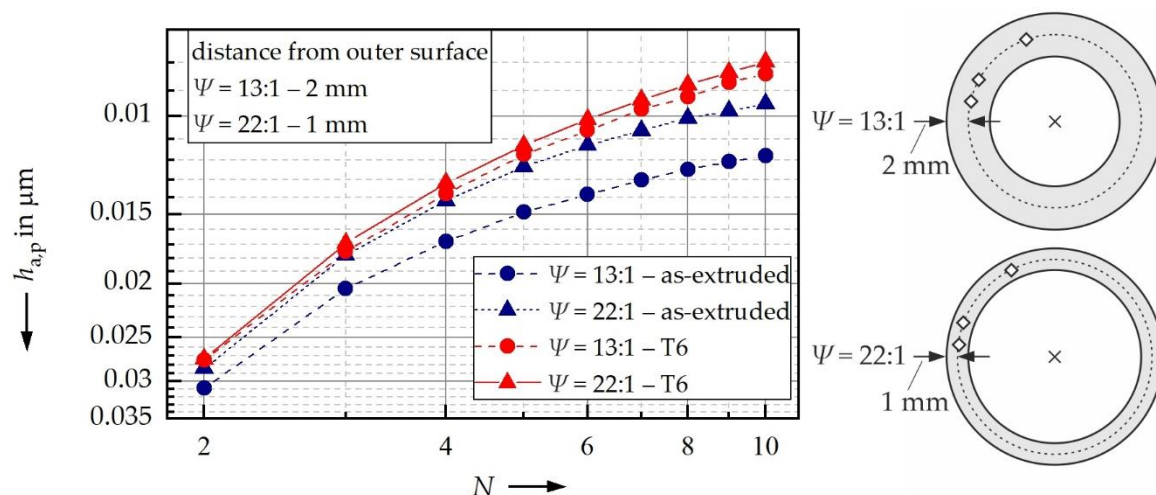


Figure 4. The $h_{a,p}$ - N curves of specimens with extrusion ratios Ψ of 13:1 and 22:1 in as-extruded state as well as after T6 heat treatment obtained in CITs in the center of the specimens' wall sections.

3. Results

Figure 4 shows the $h_{a,p}$ - N curves of all variants, i.e., the four combinations of heat treatment and extrusion ratios described in Section 2, obtained in CITs in the center of the respective specimen's wall section. Note that the vertical axis of Figure 4 is shown in reverse direction. The $h_{a,p}$ - N curves reveal significant differences between the investigated variants. In general, for both, the as-extruded and the heat-treated conditions, the higher extrusion ratio $\Psi = 22:1$ led to lower $h_{a,p}$ and, thus, a lower cyclic plasticity compared to $\Psi = 13:1$. This difference was more pronounced for the as-extruded state, which further showed, as expected, a higher cyclic plasticity than the T6 condition. Therefore, the $h_{a,p}$ - N curves demonstrated significant differences obtained by varying the extrusion ratio and applying an additional T6 heat treatment.

For a deeper understanding of the local deformation behavior, resulting from extrusion ratio and subsequent heat treatment, in the following sections the results obtained in CITs are analyzed considering the distance to the outer and inner surface and compared with the local microstructure observed by optical microscopy.

3.1. Influence of Extrusion Ratio on Hardness and Cyclic Hardening Potential

The influence of extrusion ratio on hardness (HM1/6/0.1) and cyclic hardening potential, represented by $|e_{II}|$, of the as-extruded condition is illustrated in Figure 5. For both extrusion ratios the hardness increased from the outer surface until reaching a maximum in the center of the wall, followed by a decrease to the inner surface of the Al tube (see Figure 5a,b). Comparing the different extrusion ratios, $\Psi = 22:1$ revealed higher hardness, whereas a more pronounced difference between minimum and maximum hardness was found for $\Psi = 13:1$. Yet, the hardness increase from the outer surface to the center of the wall amounted to 4% for $\Psi = 13:1$ and 1.4% for $\Psi = 22:1$ and, thus, was relatively small.

The cyclic hardening exponent $_{CHT} |e_{II}|$ attained maximum values in the peripheral region near the outer surface for both variants. With increasing distance from the outer surface d , $|e_{II}|$ decreased until reaching a minimum in the region of maximum hardness, followed by insignificant changes of $|e_{II}|$ with increasing d until reaching the inner surface. In relation to $\Psi = 22:1$, the variant with the lower extrusion ratio $\Psi = 13:1$ had a lower minimum of $|e_{II}|$ and a greater variation of $|e_{II}|$ over the entire cross section, which accorded with the more pronounced differences in hardness.

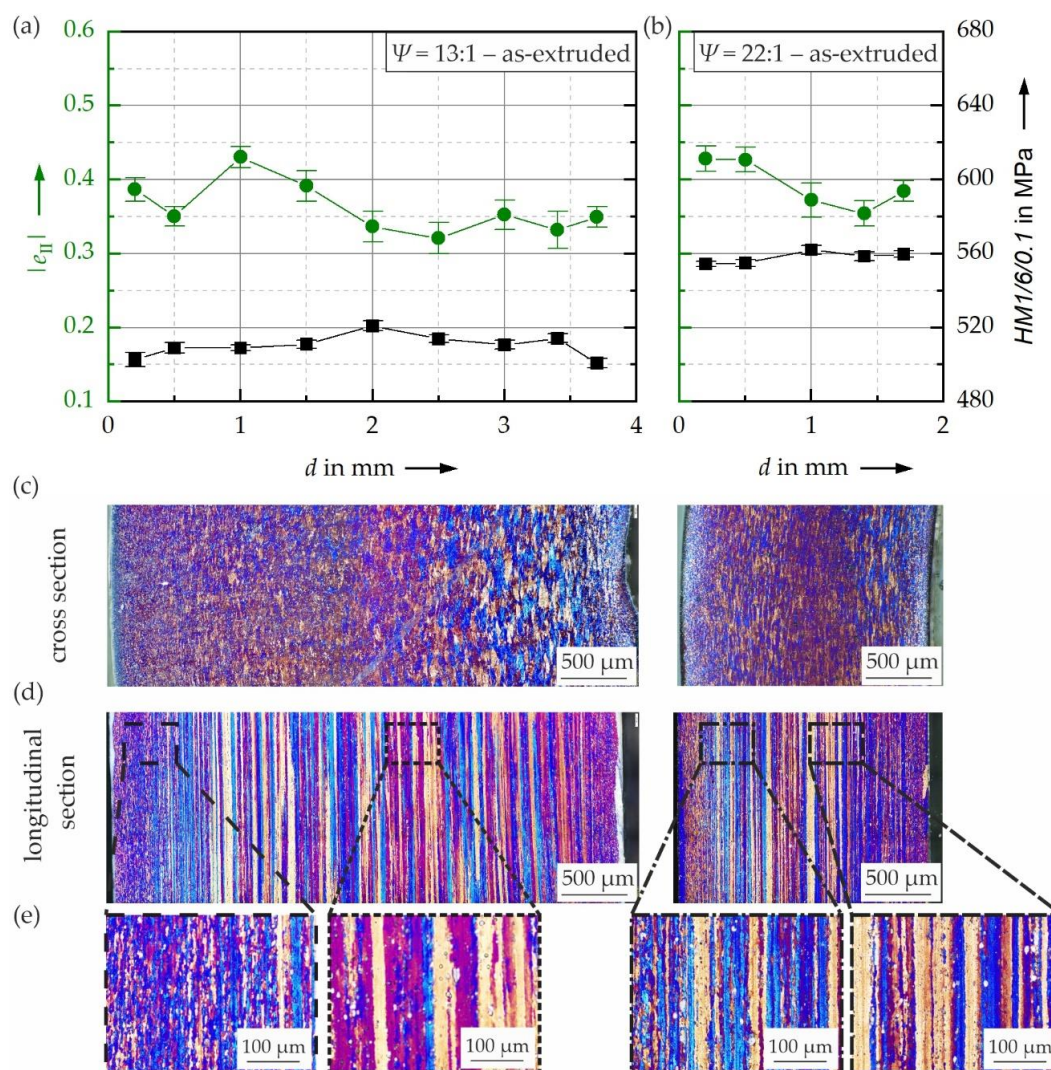


Figure 5. Martens hardness $HM1/6/0.1$ and cyclic hardening exponent $|e_{II}|$ of as-extruded state with (a) $\Psi = 13:1$ and (b) $\Psi = 22:1$, depending on the distance d from the outer surface; light optical micrographs of (c) cross sections and (d) longitudinal sections as well as (e) close-up views.

In accordance with the local mechanical properties obtained from CITs, the microstructure also showed local differences resulting from the extrusion process, which are illustrated in Figure 5c–e. A qualitative comparison showed that the microstructure in the center of the specimens consisted mainly of relatively large grains, which were elongated in the extrusion direction, while the areas near the outer and inner surface were characterized by a fine-grained zone, as clearly visible in Figure 5e.

3.2. Influence of Heat Treatment on Hardness and Cyclic Hardening Potential

As demonstrated in Figure 6a,b, applying a T6 heat treatment to the extruded EN AW-6082 tubes had a significant influence on the local mechanical properties. For both extrusion ratios, hardness and cyclic hardening exponent $|e_{II}|$ were significantly increased after heat treatment. However, the hardness profile was similar to the samples in the as-extruded conditions with an increase of hardness from the subsurface regions to a maximum in the center of the specimens, which was more pronounced for an extrusion ratio of $\Psi = 22:1$. Note that in contrast to the results obtained for the as-extruded states, the T6 heat treatment led to similar hardness for both extrusion ratios.

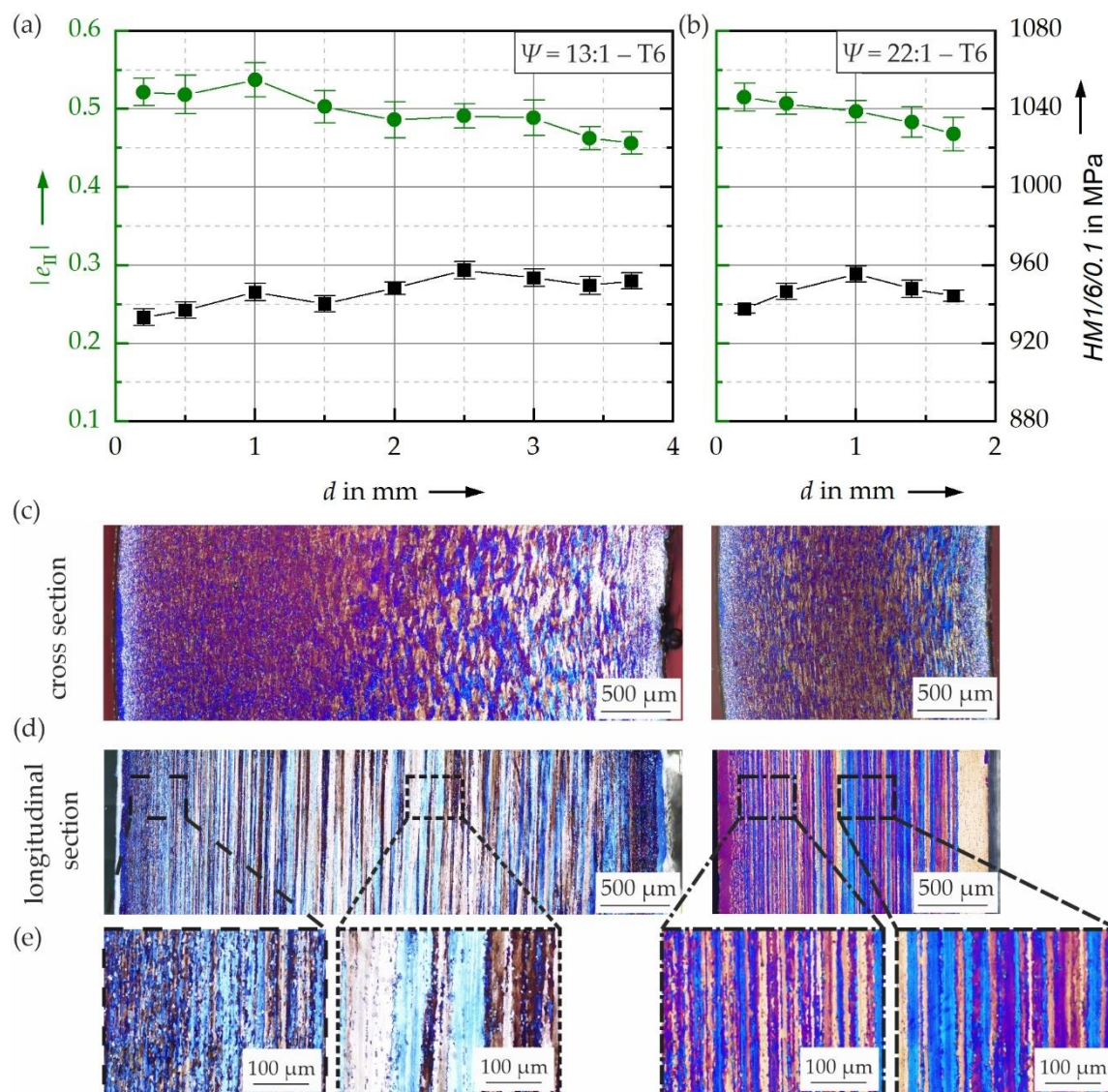


Figure 6. Martens hardness $HM1/6/0.1$ and cyclic hardening exponent_{CHT} $|e_{II}|$ of T6 heat-treatment state with (a) Ψ of 13:1 and (b) Ψ of 22:1 depending on the distance d from the outer surface; light optical micrographs of (c) cross sections and (d) longitudinal sections as well as (e) close-up views.

Considering the cyclic hardening potential, for both extrusion ratios, the difference between maximum and minimum $|e_{II}|$ was less pronounced after the T6 heat treatment. In correspondence with the as-extruded state, $|e_{II}|$ had maximum values near the outer surface and decreased with approaching the inner surface. In the case of an extrusion ratio of $\Psi = 13:1$, a plateau was reached in the center of the specimen, followed by a further decrease in the region of the inner surface. In contrast to that, the extrusion ratio of $\Psi = 22:1$ resulted in a continuous decrease of $|e_{II}|$ from the outer to the inner surface without showing a plateau in the center. Because of the greater width of Al tubes with $\Psi = 13:1$, more CITs could be placed within the cross section, which resulted in a higher spatial resolution regarding the influence of the distance to the outer surface. This might be the reason why, in contrast to $\Psi = 13:1$, no plateau was observed for $\Psi = 22:1$.

Comparing qualitatively the microstructure of the T6 heat-treated samples in Figure 6c–e with the one of the as-extruded states showed that the grain sizes of the T6 heat-treated state and the one of the as-extruded state were similar for both extrusion ratios. Thus, the heat treatment seemed to have no significant effect on the grain sizes.

4. Discussion

As presented in Section 3, both the extrusion ratio and the subsequent T6 heat treatment affected the mechanical properties of the investigated Al tubes. Based on the data obtained, these property variations can be partially explained. For both the as-extruded and the T6 heat-treated states a fine-grained zone occurred in the area of the outer and inner surface, whereas the center of the wall was characterized by relatively large and elongated grains. This can be explained by dynamic recrystallization, which occurred during the extrusion process and took place at the inner and outer surface of the tube, where the process temperature was assumed to be maximal. This was caused by friction in the extrusion tool, consisting of the die and the mandrel part (Figure 1), and was described by Ostermann [10] as being typical for AlMgSi alloys. As the grains in the center of the wall were elongated in the extrusion direction, it was assumed that no recrystallization occurred in the center of the wall. In case of the as-extruded states, this was in accordance with the cyclic hardening exponent $|e_{II}|$, which was larger in the subsurface regions due to the smaller grains, which promote cyclic hardening, as shown by Kramer et al. [15]. In contrast, the larger grains in the center led to a smaller $|e_{II}|$.

However, the grain size distribution suggests that hardness was maximum in the subsurface region due to the smaller grain sizes, which was opposite to the experimental data obtained, showing maximum hardness in the center. Therefore, a superposition of different microstructural phenomena had to be assumed and other influencing factors, such as dislocation density, had to be considered for a complete understanding of the mechanical properties. Additionally, the variations of grain sizes seemed to have less impact on mechanical properties, which could also be seen by the similar amount of scatter at different positions of the tubes despite different grain sizes. A possible explanation for the presented observations is a reduction of dislocation density in the subsurface regions caused by recrystallization during the extrusion process, which would lead to a reduction of hardness [8,9]. Moreover, the higher hardness and lower plasticity of variant $\Psi = 22:1$ might be explained by the generally higher dislocation density, resulting from the higher extrusion ratio and, thus, more pronounced plastic deformation during the manufacturing process. In this context, other investigations had already demonstrated unequivocally that extrusion parameters have an impact on dislocation density [8,9]. However, the determination of dislocation density was not within the scope of the presented study. To verify these assumptions, microstructural investigations with higher resolution, i.e., transmission electron microscopy (TEM) or X-ray diffraction (XRD), are required.

As shown in Section 3.2 the T6 heat treatment significantly influenced the local mechanical properties, which cannot be explained by a change in grain size, being similar between the as-extruded and T6 heat-treated conditions. Therefore, other microstructural characteristics were assumed to have occurred, which led to these changes. The solution annealing can be assumed to have resulted in a decrease of dislocation density, whereas the subsequent artificial aging led to the formation of precipitates, as shown by [27,28]. Note that precipitation hardening can be assumed to result in an increased hardness and cyclic hardening potential, which correlates with the results obtained (Figure 4) [17,23,25]. As shown in a vast number of previous studies, a T6 heat treatment leads to a strength increase of Al alloys by precipitation hardening, see, e.g., [29,30]. Thus, the precipitation state as well as the dislocation density have to be considered for explaining the changes observed in mechanical properties. However, this requires high-resolution microscopy and is the objective of further investigations.

The presented results showed that both, the extrusion ratio and a T6 heat treatment, had a significant influence on hardness and cyclic hardening potential of extruded Al EN AW-6082 tubes. By using CITs, these differences were determined with high lateral resolution within the tubes, showing local differences of deformation behavior within the cross sections of the analyzed tubes. As demonstrated in [18], knowing only hardness is not sufficient to assess the mechanical properties and especially cyclic properties, i.e., the fatigue behavior. Determining also the cyclic hardening potential enables a better

assessment of fatigue properties, which makes CITs superior to conventional hardness tests. However, for a complete understanding of the obtained mechanical properties, further extensive investigations based on high-resolution microscopy are indispensable to verify the proposed hypothesis regarding the interrelation of extrusion process, dislocation density, precipitation state, and resulting cyclic deformation behavior.

5. Conclusions

In this work, the mechanical properties of extruded tubes made of EN AW-6082 were investigated by using cyclic indentation tests (CITs) and the results obtained were compared with the local microstructure analyzed by light optical microscopy. To determine the influence of deformation during the manufacturing process, extrusion ratios Ψ of 13:1 and 22:1 were used. Moreover, the impact of a T6 heat treatment on the microstructure and the mechanical properties was examined by comparing the results obtained in the extruded state and after a T6 heat treatment. The CITs enabled a local analysis of the mechanical properties and, thus, their dependency on the distance to the outer surface of the EN AW-6082 tubes. The main findings can be summarized as follows:

- Hardness and cyclic hardening potential varied within the cross section for both the as-extruded and the T6 heat-treated states. This can be associated with differences in microstructure.
- In the as-extruded state, increasing the extrusion ratio from 13:1 to 22:1 led to an increase of hardness and decrease of cyclic plasticity, which nearly disappeared when applying a subsequent T6 heat treatment.
- A T6 heat treatment increased the hardness and the cyclic hardening potential and led to a decrease of cyclic plasticity obtained in CITs.
- The resulting mechanical properties cannot be explained solely based on local grain morphology, as other influencing factors such as dislocation density or precipitation state are assumed to have a significant impact as well.
- It was shown that CITs can be used to determine the local cyclic properties of extruded Al alloy tubes.

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