Processing induced size effects in plastic yielding upon miniaturisation

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Abstract

Size effects in metals have received considerable attention in literature in the last decades. For preparing specimens dedicated processing techniques, such as laser-cutting, micro-milling, turning, etc., are used. Most of these processing methods intrinsically damage crystals just below the worked surface. In macroscopic applications, the effect on the overall mechanical behaviour can safely be neglected in most cases. Upon miniaturisation, however, the influence of the affected region becomes more important and may induce a processing induced size effect, which is far from negligible. Processing induced size effects are analysed by carefully characterising the plastic yielding in uniaxial tension of rectangular, 300 μm-thick aluminium sheet specimens, with a well-defined homogeneous microstructure containing through-thickness grains. The specimens are processed to different widths by three independent machining techniques: (1) laser-cutting, (2) mechanical cutting, and (3) extensive grinding from a larger width. These independent techniques all result in a distinct processing induced size effect upon miniaturisation, i.e. an increase of up to 200% in yield stress for a decrease from about 12 to 3 grains over the specimen width. Using a simple Taylor averaging model, it is shown that the yield stress in the affected edge region increased to 210–350% of its initial (or bulk) value. In addition, it is found that even a prolonged anneal near the melting temperature can only partially remove the processing induced size effect. The results clearly demonstrate that processing induced size effects have to be considered in the design of miniaturised devices and parts as well as in scientific research relying on the testing of manufactured small-scale test specimens.

Keywords: Size effects; Microstructures; Crystal plasticity; Metallic materials; Mechanical testing

1. Introduction

Miniaturisation reflects the drive towards the design and manufacturing of smaller and smaller devices, components, and parts. Examples in which this miniaturisation plays a significant role include micro-electronics devices (for example RF-MEMS; micro-grippers, micro-mirrors, and switches) and (bio-)medical applications (for example cardiovascular stents) (Connolley et al., 2005; Kathuria, 2005). The state-of-the-art

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in micro-manufacturing technologies (e.g. turning, grinding, micro-milling, micro-punching, electro-discharge machining, laser-cutting, etc.) worldwide is presented in the World Technology Evaluation Center (WTEC) Panel report (2005). When the component’s dimensions are decreased to the same order of magnitude as the material’s microstructural length scales, e.g. the grain size, significant changes in mechanical properties may occur, commonly denoted as 'size effects'. If ignored, these size effects may lead to erroneous engineering assessments or designs.

For the plastic deformation of metals, at least four size effects have been suggested in the literature : (i) strengthening or weakening due to constrained or free boundary layers, (ii) strengthening due to strain gradients, (iii) strengthening due to dislocation starvation, and (iv) size effects due to a lack of statistical microstructural averaging. The first size effect results from the formation of a strong or weak boundary layer at a surface, because the slip of dislocations is blocked or facilitated resulting in pile-up or depletion of dislocations near the boundary and consequently in an increased or decreased resistance against further plastic flow (e.g. Miyazaki et al., 1979; Arzt, 1998; Kals, 1998; Janssen et al., 2006; Geers et al., 2006). The strengthening or weakening behaviour of a surface boundary layer has also been reported by Han et al. (2006) using discrete dislocation dynamics simulations. As the geometrical dimensions are reduced, the influence of the boundary layers becomes more pronounced and the deviation from classical continuum mechanics theory increases. Note that an unpassivated surface covered with a native oxide is not necessarily a free surface. Similar constraints may occur at internal grain boundaries, giving rise to a grain-size dependence cf. the Hall–Petch effect (Hall, 1951; Petch, 1953; Armstrong et al., 1962), or at the surface of second-phase particles (Arzt, 1998). The second size effect is attributed to the presence of strain gradients (e.g. Fleck and Hutchinson, 1997; Stölken and Evans, 1998; Shu et al., 2001; Evers et al., 2004; Nicola et al., 2005; Bayley et al., 2006; Geers et al., 2006), for instance during bending, resulting in inhomogeneous plastic deformation which in turn is accomplished by the presence and evolution of geometrically necessary dislocations (GNDs). GNDs represent the similarly signed fraction of all dislocations, contributing to slip and flow resistance as any other dislocation, except for the additional contribution of a back-stress resulting from the dislocation polarity. For equal strain levels, a smaller foil thickness implicates a higher strain gradient and therefore a higher GND density. A third origin of size effects has been observed in (sub)micron-sized, near-defect-free structures (Greer et al., 2005). Though still subject of discussion, the observed increase in resistance against plastic flow in these structures has been attributed to dislocation starvation, i.e. dislocations flow out easily upon loading, thus drastically limiting the number of dislocations available for further slip and thereby strengthening the material. Finally, a special case of size effects is found in polycrystalline (or multi-crystalline) metals upon decreasing (geometrical) dimensions of a structure (Fülöp et al., 2006; Henning and Vehoff, 2007; Bayley et al., 2007). As a result of limited averaging over the local microstructural variations, the mechanical behaviour of the overall structure becomes dependent on the detailed underlying microstructure (e.g. size, distribution, and orientation of the individual grains). This effect appears most significantly for structures with dimensions close to the characteristic microstructural length scale, e.g. only a few grains in a spatial direction, where the material response is governed by the weakest link (Armstrong et al., 1962; Henning and Vehoff, 2007). This typically induces a weaker response for smaller structures, accompanied with an increase in the statistical variation in the observed engineering strength (Fülöp et al., 2006; Bayley et al., 2007). Note that for most cases where microparts are made top-down from a base material, this size effect appears first if the microstructure remains unaffected.

Whereas the above-mentioned size effects have received considerable attention in the literature, size effects upon miniaturisation caused by the adopted processing method of microparts seem to be largely underestimated, both in the industry and scientific community. In the industry, various manufacturing techniques are used to produce (miniature) components and parts, such as laser-cutting (Araújo et al., 2003; Fu et al., 2006), blanking (Maiti et al., 2000), micro-milling (Vogler et al., 2004; Okazaki et al., 2004), turning (To and Lee, 2001; Jaspers and Dautzenberg, 2002; M’Saoubi and Ryde, 2005) and grinding (Okazaki et al., 2004; Ohmori et al., 2003). It is well known, since long, that these manufacturing techniques intrinsically introduce damage in crystals just below the affected surface, i.e. a zone will exist with different mechanical properties compared to the ‘bulk’ properties. In macroscopic applications, the influence of the affected volume can be safely neglected compared to the total volume of the part. Upon miniaturisation, however, the relative influence of the damaged zones becomes more important, possibly causing significant changes in the
mechanical response of the overall component, which will therefore be denoted here as a ‘processing induced size effect’. In scientific research small-scaled specimens are often used to analyse a material’s intrinsic mechanical behaviour, e.g. as done in the experimental investigations of the above-mentioned size effects (i)–(iv). Care must be taken to avoid that such analyses are influenced by processing induced size effects.

The aim of the present work is to analyse processing induced size effects. This research will be performed on well-defined test specimens with a microstructural length scale similar to the geometrical length scale. Note that these specimens, with through-thickness grains and only a limited number of grains across the width, are geometrically similar to the smaller RF-MEMS structures typically used in the electronic industry. To analyse the influence of the applied manufacturing technique on the processing induced size effects, the test specimens are produced using three independent manufacturing methods: (1) laser-cutting, which is a commonly used micro-manufacturing technique (e.g. Connolley et al., 2005; Kathuria, 2005; Fu et al., 2006; WTEC Panel report, 2005); (2) mechanical cutting, which is a shear-dominated separation process as used in micro-stamping, micro-punching, and fine-blanking applications (e.g. Metal Industries Research & Development (MIRDC) Research report, 2004; Joo et al., 2001; Maiti et al., 2000; WTEC Panel report, 2005), and (3) extensive grinding from a larger width (e.g. Okazaki et al., 2004; Ohmori et al., 2003).

The effect of these processing methods on the mechanical response is analysed by tensile experiments on specimens with different widths (2–10 mm). In this analysis other possible size effects have been excluded by making motivated choices for the selection and conditioning of the specimens, the base material, and the mechanical testing applied. It will be emphasised that processing induced size effects in plastic yielding upon miniaturisation are far from neglectable. The originality of this work results from:

- The rigorous analysis of the size effect of interest.
- The generic implication that is put forward, i.e. these effects are expected to be present for any processing method and they are increasingly important if the size reduces. Yet, quantitative differences will result depending on the method used.
- The analysis carried out in view of the possible reduction of this size effect with subsequent annealing treatments.

The results clearly demonstrate the importance of taking processing induced size effects into account in the design process of micro devices and scientific research.

2. Experimental procedure

2.1. Material selection

A high purity material is selected to exclude dominant contributions of complex microstructural deformation modes related to the presence of second phase particles. A metal with a face centered cubic (FCC) structure is preferred. The slip systems for FCC metals are well defined and their deformation is generally well understood. Moreover, FCC metals are commonly used and e.g. play a significant role in thin film MEMS structures. A high stacking fault energy is preferred because this hampers the formation of twins. For these reasons, high purity aluminium (99.999 at%, Goodfellow) is used. The original aluminium sheets, 150 mm × 150 mm, are received in ‘as-rolled’ condition with a thickness of 310 μm and an average grain size of approximately 100 μm, see Fig. 1. The microstructure was analysed using an FEI Sirion scanning electron microscope, equipped with a TexSem Laboratories orientation imaging detector (OIM), see for specimen preparation Janssen et al. (2006).

2.2. Specimen preparation

The as-received Al sheet material is further processed to create a ‘base material’ with a well-defined homogeneous microstructure of through-thickness grains, see Fig. 2. All grains have at least two free surfaces, i.e. small variations in grain size do not affect the mechanical properties significantly (Janssen et al., 2006). The base material is prepared, using the following procedure.
Strips of 150 mm × 30 mm were mechanically cut from the as-received Al sheets parallel to the original rolling direction. After cutting, the material was annealed for 1 h at 200 °C. Next, the strips were uniaxially pre-strained to 5.5% strain, to create the required nucleation sites for recrystallisation. The final microstructure was obtained by a recrystallisation heat treatment of 30 min at 600 °C (more details about the preparation procedure is presented below). A reproducible, homogeneous microstructure with an average grain size of 800 μm (area averaged, measured on the specimen surface) and a pronounced cube texture is produced using this strain-anneal protocol. Detailed microstructural analyses, examining longitudinal cross-sections, showed that less than 8% of the grains are not through the thickness; these grains occupy only about 1.4% of the cross-sectional area of the specimens and, therefore, their influence on the overall mechanical behaviour of the specimen is neglected. Note that the recrystallisation removed all prior mechanical cutting effects, as confirmed through nano-indentation measurements.

Three specimen types are prepared from the base material. Specimens were shaped to their final geometry by (1) laser-cutting, (2) mechanical cutting, and (3) extensive grinding from a larger width. Furthermore,
specimen sets are prepared with additional treatments steps of moderate grinding or annealing. An overview of the prepared specimens are presented in Tables 1–3.

The laser-cut specimens (Table 1), with a length of 40 mm, were cut from a central 100 mm × 30 mm region of the 150 mm × 30 mm (base material) strips, preserving a distance of 1 mm from the edges. A Lasag CO₂ laser was used at a standard oxygen pressure of 20 bar, spotsize of 70 μm, frequency of 120 Hz, pulse length of 0.2 ms and voltage of 350 V. This resulted in a cutting energy of 211 mJ per pulse.

In the mechanical cutting process (Table 2), a Gerver corner shear (type: GH-1) apparatus is used. Two smaller strips of 50 mm × 30 mm were cut from the central area of a 150 mm × 30 mm (base-material) strip. Thereafter, specimens, with a length of 50 mm, were cut from the 50 mm × 30 mm strips. This was done by

Table 1
Laser-cut specimen sets ((#) indicates the number of specimens tested)

<table>
<thead>
<tr>
<th>Specimen set (#)</th>
<th>Final width (mm)</th>
<th>Annealing I</th>
<th>Annealing II</th>
</tr>
</thead>
<tbody>
<tr>
<td>L_A (2)</td>
<td>9.6</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>L_A (2)</td>
<td>4.1</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>L_A (2)</td>
<td>2.1</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>L_B (1)</td>
<td>9.6</td>
<td>1 h at 200°C</td>
<td>–</td>
</tr>
<tr>
<td>L_B (2)</td>
<td>4.1</td>
<td>1 h at 200°C</td>
<td>–</td>
</tr>
<tr>
<td>L_B (2)</td>
<td>2.1</td>
<td>1 h at 200°C</td>
<td>–</td>
</tr>
<tr>
<td>L_C (2)</td>
<td>9.6</td>
<td>1 h at 200°C</td>
<td>0.5 h at 600°C</td>
</tr>
<tr>
<td>L_C (2)</td>
<td>4.1</td>
<td>1 h at 200°C</td>
<td>0.5 h at 600°C</td>
</tr>
<tr>
<td>L_C (2)</td>
<td>2.1</td>
<td>1 h at 200°C</td>
<td>0.5 h at 600°C</td>
</tr>
</tbody>
</table>

Table 2
Mechanically cut specimen sets ((#) indicates the number of specimens tested)

<table>
<thead>
<tr>
<th>Specimen set (#)</th>
<th>Initial width (mm)</th>
<th>Edge grinding</th>
<th>Final width (mm)</th>
<th>Annealing I</th>
<th>Annealing II</th>
</tr>
</thead>
<tbody>
<tr>
<td>M_A (3)</td>
<td>–</td>
<td>n</td>
<td>8.9</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>M_A (4)</td>
<td>–</td>
<td>n</td>
<td>2.1</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>M_B (4)</td>
<td>12.0</td>
<td>y</td>
<td>9.4</td>
<td>1 h at 200°C</td>
<td>–</td>
</tr>
<tr>
<td>M_B (5)</td>
<td>4.0</td>
<td>y</td>
<td>2.3</td>
<td>1 h at 200°C</td>
<td>–</td>
</tr>
<tr>
<td>M_C (2)</td>
<td>12.0</td>
<td>y</td>
<td>9.5</td>
<td>1 h at 200°C</td>
<td>20 h at 200°C</td>
</tr>
<tr>
<td>M_C (3)</td>
<td>4.0</td>
<td>y</td>
<td>2.3</td>
<td>1 h at 200°C</td>
<td>20 h at 200°C</td>
</tr>
<tr>
<td>M_D (3)</td>
<td>12.0</td>
<td>y</td>
<td>9.5</td>
<td>1 h at 200°C</td>
<td>8 h at 600°C</td>
</tr>
<tr>
<td>M_D (3)</td>
<td>4.0</td>
<td>y</td>
<td>2.3</td>
<td>1 h at 200°C</td>
<td>8 h at 600°C</td>
</tr>
<tr>
<td>M_as-received (3)</td>
<td>–</td>
<td>n</td>
<td>8.8</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>M_as-received (5)</td>
<td>–</td>
<td>n</td>
<td>2.2</td>
<td>–</td>
<td>–</td>
</tr>
</tbody>
</table>

Table 3
Extensively ground specimen sets ((#) indicates the number of specimens tested)

<table>
<thead>
<tr>
<th>Specimen set (#)</th>
<th>Cutting</th>
<th>Initial width (mm)</th>
<th>Extensive grinding</th>
<th>Final width (mm)</th>
<th>Annealing I</th>
</tr>
</thead>
<tbody>
<tr>
<td>G_A (2)</td>
<td>Mechanical</td>
<td>14.0</td>
<td>y</td>
<td>9.0</td>
<td>–</td>
</tr>
<tr>
<td>G_A (3)</td>
<td>Mechanical</td>
<td>14.0</td>
<td>y</td>
<td>2.0</td>
<td>–</td>
</tr>
<tr>
<td>G_B (2)</td>
<td>Laser</td>
<td>9.6</td>
<td>y</td>
<td>8.5</td>
<td>1 h at 200°C</td>
</tr>
<tr>
<td>G_B (2)</td>
<td>Laser</td>
<td>4.1</td>
<td>y</td>
<td>2.3</td>
<td>1 h at 200°C</td>
</tr>
</tbody>
</table>
translating parts (i.e. specimen) of these strips after every cut under the shearing knife until the complete strip was cut, discarding an edge region of 1 mm on both sides. In the cutting process, the uncut part of the base strip was placed under a blank holder, while the specimen under the knife was supported by a manually controlled cushion, i.e. comparable to a low-pressure cushion used in fine blanking. The difference in specimen length between laser-cut and mechanically cut specimens was only due to a maximum length that could be obtained by laser-cutting.

For the third method (Table 3), i.e. extensive grinding from a larger width, the edges of 14 mm wide mechanically pre-cut specimens were ground down to decrease the specimen width to 9 and 2 mm, respectively. For the edge grinding, the specimens were carefully positioned in a holder. The grinding was done on Struers SiC paper with grit sizes of 800, 1200, 2400, and 4000 (FEPA), having grain sizes of 21.8, 15.2, 10 and 5 μm, respectively, using water as a coolant and lubricant. Each grinding step was continued till the surface damage introduced by the previous step was completely removed and thus replaced by the surface damage introduced by the next SiC paper. The grinding direction for the 800 and 1200 grit paper was perpendicular to the specimen edges and for the 2400 and 4000 SiC paper parallel with the specimen edges. A similar protocol was used for the laser pre-cut specimens (see Table 3).

The annealing treatments were done in a Carbolite CSF 1200 furnace. Before positioning the material or specimens, the chamber temperature was stabilised at the required level for several hours. After annealing, the specimens (naturally) cooled down in air to room temperature outside the furnace.

2.3. Tensile tests

The basic mechanical behaviour is measured via uniaxial tensile experiments, which is a macroscopically homogeneous deformation method, whereby macroscopic strain gradient effects can be neglected in first instance. The measurements were performed at room temperature using a Kammrath&Weiss 10 kN micro-loading stage equipped with a 100 N loadcell. The displacement and strains were measured on the specimen surface by a digital image correlation technique (e.g. Chu et al., 1985) (Aramis, www.gom.com). The gauge length was 30 mm for the laser-cut specimens and 35 mm for the mechanically cut specimens. It has been verified experimentally and numerically that a length to width ratio of at least 3 for the gauge length is sufficient to exclude undesired clamping effects. The tensile tests were displacement controlled and the strain rate was 5.0 × 10⁻⁴/s. The engineering stresses, σ, and logarithmic strains, ε, were calculated from the measured force, F, and elongation using: 

\[ \varepsilon = \ln \left( \frac{l}{l_0} \right) \quad \text{and} \quad \sigma = F/A_0, \]

where \( l \) represents the actual length, \( l_0 \) the initial length and \( A_0 \) the initial cross-sectional area. The tensile curves are analysed for strains up to 0.05 (absolute strain), focussing on the onset of plastic yielding. The yield stress is determined through the engineering stress at an offset strain of 0.002 (generally known as \( R_{p0.2\%} \)). Experimental deviations, such as misalignment of the specimens, clamping effects and the tensile set-up itself, have been evaluated and were proven to be insignificant for the results obtained. It was furthermore verified that there was no significant recovery of the specimens at room temperature.

2.4. Nano-indentation

Nano-indentation experiments were done using a Berkovich indentor in an MTS Nanoindentor XP. The tests were displacement controlled, with a maximum indentation depth of 3 μm at an indentation rate of 50 nm/s. In this study, the force versus indentation depth is measured and analysed. One of the main reasons to focus on the indentation force and not on the hardness, resides in the fact that the contact surface is often ill-defined. This particular topic has been addressed by various authors in the literature (e.g. Widjaja et al., 2007), and therefore it is preferred to use the ‘raw’ force data instead, which is not debatable in this sense. Each measurement set consists of four parallel scans across the width of a specimen, with a spacing of 100 μm. The distance between the indents within a single scan across the specimen width was 100 μm in the edge region and 200 μm in the specimen centre. From these parallel scans, an average indentation force at a specific distance from the specimen edge is calculated. All specimens surfaces were electro-polished (cf. Janssen et al., 2006) before the nano-indentation measurements, eliminating variations in (native) oxide layers. Nano-indentation experiments performed on the top and bottom surfaces of the same specimen did not reveal any significant
differences in the results. Note that fluctuations in the indentation force occur due to the orientation differences of the indented crystals and the relative position of the indentation site with respect to grain boundaries. In spite of all precautions taken, random changes in absolute indentation forces due to specimen mounting and laboratory temperature shifts still prevented a quantitative inter-specimen comparison of the absolute indentation force. Nevertheless, a relative comparison of the indentation forces on a single specimen remained well possible.

3. Results and discussion

3.1. Identification of processing induced size effects

3.1.1. Processing induced size effects for laser-cut and mechanically cut specimens

The results for the laser-cut and mechanically cut specimens are investigated first. The uniaxial tensile responses for the laser-cut specimens in as-cut condition, set LA, are presented in Fig. 3a and the resulting yield stresses (and their standard deviations\(^1\)) are listed in Table 4. The narrow, 2.1 mm wide, specimens present a significantly higher stress–strain response in the low-strain region compared to the 9.6 mm wide specimens. The 4.1 mm wide specimens show an intermediate behaviour. The stress–strain responses of the mechanically cut specimens in as-cut condition, set MA, are shown in Fig. 3b. The narrow specimens again reveal significant strengthening. Both the laser-cut and the mechanically cut specimens show a considerable increase in the yield stress, close to a factor 2 for the considered decrease in width.

This observed ‘narrow is stronger’ effect may result from various effects (see e.g. Arzt, 1998), which are considered below. The effect of the oxide layer was investigated by comparing the tensile behaviour of specimens with different oxide layers. To this purpose, different annealing treatments were used, with optional removal of the oxide layer in an HF solution. The measured yield stress did not depend on the presence or type of oxide layer for specimens of the considered size. In addition, the grain structure and texture of the laser-cut and mechanically cut specimens were characterised after processing, as shown in Fig. 4 for laser-cutting. Clearly, the laser-cutting process does not introduce any differences or changes in the grain structure near the edges of the specimen, also systematic differences in texture between the edge and bulk grains cannot be observed. Similar conclusions hold for the mechanically cut specimens.

Free surface effects do not contribute to the observed ‘narrow is stronger’ behaviour. Grains at the edges are kinematically less constrained than their counterparts in the bulk of the material, since they have three free

\(^1\)The use of a standard deviation in the present context is questionable because of the limited number of measurements. However, it is here only used to give an indication of the statistical variation in the results.
surfaces whereas the bulk grains have only two free surfaces. Dislocations can escape more easily from these edge grains than from bulk grains. The weakening effect is typically opposite to the Hall–Petch effect, where dislocations are obstructed by grain boundaries causing an increase in yield stress. Note that the initial yield stress considered is an engineering yield stress, at which plastic flow is already quite active at the scale of individual dislocations. As a result, a smaller force is normally required to deform the edge grains. The volume fraction of edge grains is significantly higher for the narrow specimens than for the broad specimens. Thus, free surface effects would induce a decrease in engineering yield stress with decreasing width, which is opposite to the results shown in Fig. 3.

Grain statistics are also not responsible for the observed size effect. For both narrow and broad specimens, the mechanical behaviour will deviate from that of the bulk material due to a limited averaging of the mechanical properties of the individual grains. The material behaviour and especially the yield stress will be determined by chains of weak grains covering the full width of the specimen, resulting in a weaker overall response (e.g. Armstrong et al., 1962). This effect should be more pronounced for narrow specimens that have about 3 grains across to width compared to the 12 grains for the broad specimens.

The only physically meaningful explanation for the observed difference in response of the narrow and broad specimens is the occurrence of a processing induced size effect in plastic yielding. It is generally known that processing alters local material properties, e.g. for laser-cutting a heat affected zone is usually present (Araújo et al., 2003; Kals and Eckstein, 2000; Yilbas, 2004). Yet, the influence for miniaturised specimens turns out to be larger than expected. To investigate the affected edge zone, nano-indentation experiments are conducted to measure possible variations in mechanical properties across the width of the specimens. In Fig. 5, the indentation forces at several indentation depths are shown for a broad and narrow laser-cut specimen, in as-cut condition.

All specimens reveal a progressively increasing indentation force towards the edge, the relative magnitude of which remains approximately independent of the indentation depth. The size of the edge zone is here defined

Table 4
Yield stress ($\sigma_y$) and its standard deviation in MPa, for specimens of different sets and widths

<table>
<thead>
<tr>
<th>Specimen set</th>
<th>Conditioning</th>
<th>Specimen width</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>~9 mm</td>
</tr>
<tr>
<td>L_A</td>
<td>as-cut</td>
<td>7.4 (0.4)</td>
</tr>
<tr>
<td>L_B</td>
<td>as-cut, 1 h 200 °C</td>
<td>6.9 (–)</td>
</tr>
<tr>
<td>L_C</td>
<td>as-cut, 1 h 200 °C, 0.5 h 600 °C</td>
<td>5.8 (0.4)</td>
</tr>
<tr>
<td>M_A</td>
<td>as-cut</td>
<td>15.0 (0.5)</td>
</tr>
<tr>
<td>M_B</td>
<td>as-cut, mg, 1 h 200 °C</td>
<td>8.6 (0.4)</td>
</tr>
<tr>
<td>M_C</td>
<td>as-cut, mg, 1 h 200 °C, 20 h 200 °C</td>
<td>7.6 (0.9)</td>
</tr>
<tr>
<td>M_D</td>
<td>as-cut, mg, 1 h 200 °C, 8 h 600 °C</td>
<td>5.4 (0.4)</td>
</tr>
<tr>
<td>M_as-received</td>
<td>as-cut</td>
<td>23.9 (0.5)</td>
</tr>
<tr>
<td>G_A</td>
<td>ext. ground</td>
<td>7.3 (0.1)</td>
</tr>
<tr>
<td>G_B</td>
<td>ext. ground, 1 h 200 °C</td>
<td>7.3 (0.1)</td>
</tr>
</tbody>
</table>

Subsequent treatments after processing are indicated in the table (mg = moderate gridding).
as the distance from the specimen edge to the position where the indentation force is higher than the average bulk value plus its standard deviation.

An average edge zone of 1.3 mm can be identified for the laser-cut specimens. Similar nano-indentation measurements for the mechanically cut specimens reveal an average edge zone width of 2.0 mm. The strength and size of the edge zone depends on the processing technique, but is independent of the specimen width. The edge zone, e.g. as induced by laser-cutting, is much larger than what would be expected on the basis of the literature. Even though specific processing conditions are different, results in literature were expected to give a good indication. Based on electron microscopy measurements a narrow heat affected zone width of a few microns has been reported (Araújo et al., 2003; Kals and Eckstein, 2000; Yilbas, 2004). The effect of such a small zone on the mechanical properties of the specimens should not be significant. Furthermore, the orientation imaging microscopy results (see Fig. 4) did not show any significant influence on the grain structure or texture of the material. The nano-indentation experiments on the contrary, clearly reveal that a much wider influenced zone exists, which also explains the resulting size effect on the tensile properties of the specimens.

To validate that the observed size effect is not specific for the currently analysed specimens with through-thickness grains, mechanically cut specimens with a smaller average grain size of 100 \( \mu \)m, set \( \text{MA-received} \), have been examined as well (i.e. the as-received material, see Section 2.1). The tensile results are shown in Fig. 6a and the yield stresses are listed in Table 4. Narrow specimens again show a stronger response, with a difference in yield stress for both specimen widths that is comparable to the mechanically cut specimens of the base material (set \( \text{MA, Table 4 and Fig. 3b} \)). The nano-indentation experiments reveal an edge zone of 1.0 mm, as shown in Fig. 6b. The higher yield stresses and hardening of these specimens is due to the smaller grain size (Hall–Petch effect) and the presence of grain boundaries parallel to the specimen's surface. During deformation, these parallel grain boundaries obstruct dislocation movement and hamper the rotation of the crystals, triggering a stronger response, as shown by (Janssen et al., 2006).

Undoubtedly, the observed ‘narrow is stronger’ effect results from the manufacturing process of the specimens, inducing an edge zone with an increasing strength towards the edges. Whereas the presence of an edge zone is well known since decades, its size and large quantitative role in miniaturised specimens is certainly striking. The volume fraction of the edge zone is significantly higher for narrow specimens than for broad specimens, resulting in a stronger initial tensile behaviour, of about 200%, of the narrow specimens compared to the responses of the broad specimens.

\(^2\)The size has been determined from measurements on the \( \sim 9 \) mm specimens because in the narrow specimens an ‘overlap’ of these zones is present.
3.1.2. Processing induced size effects for extensively ground specimens

Extensive grinding of the edges of the mechanically cut specimens, set GA, is used to analyse up to what extent the processing induced size effect, as induced by the mechanical cutting, can be removed. It is well known that the grinding itself also induces damage to the material and crystals just below the ground surface (e.g. Samuels, 1956–1957; Turley and Doyle, 1975; Wong and Doyle, 1999; Petzow, 1999). In traditional applications, this effect on the overall mechanical response is commonly neglected. Upon miniaturisation, however, this effect may still be significant, which is investigated next. The specimen preparation protocol is given in Section 2.2. Nano-indentation experiments are performed across the width of the specimens and the results are shown in Fig. 7. As can be observed, an edge region (as defined in Section 3.1.1) cannot be distinguished anymore. The edge effect as introduced by mechanically cutting has been eliminated, by grinding at least 2.5 mm of each edge. Nevertheless, near three of the four edges, shown in Fig. 7, a small gradient in the indentation force in a region of approximately 400 μm can be noticed. This may indicate that a small grinding-induced edge zone is still present, for which the indentation force would be higher in the unmeasured region close to the edge. The presence of a narrow grinding-induced zone would be consistent with a reported study of the induced subsurface deformation in brass (Samuels, 1956–1957, grain size of 400 μm), which showed that
the deformation depth is in the order of the grain size of the used SiC paper. For the 800 and 1200 grit papers used here, the deformation depth would be in the order of about 20 $\mu$m, which is likely a lower bound value because of the recrystallised pure aluminium used here.

Tensile tests have been performed on specimens from the same set, for which the results are shown in Fig. 8a. It is observed that the narrow specimens still show a stronger mechanical response, i.e. a small processing induced size effect persists. The observed difference is caused by the presence of a relatively narrow, but strong edge zone, due to the grinding of the edges. Similar to the case of laser and mechanical cutting, the volume fraction of this grinding-induced edge zone is higher for the narrow specimens, resulting in a stronger initial stress–strain response.

A similar edge grinding procedure has been applied to the laser-cut specimens. For these specimens, set G_B, most of the edge region has been removed, whereafter they were annealed for 1 h at 200 $^\circ$C (see also Table 3). The effect of the 200 $^\circ$C anneal is small as obvious from Fig. 9, discussed below. The tensile behaviour of these specimens is presented in Fig. 8b. A processing induced size effect is clearly present. Comparing the results for the extensively ground mechanically cut specimens (set G_A) and laser-cut specimens (set G_B), highlights some

![Image](image1)

Fig. 8. Stress–strain curves of (a) extensively ground mechanically pre-cut specimens (set G_A) and (b) extensively ground laser pre-cut specimens annealed for 1 h at 200 $^\circ$C (set G_B).

![Image](image2)

Fig. 9. Effect of grinding the specimen’s surface and the influence of subsequent annealing treatments on the indentation force. Each curve is averaged over ~20 indentation curves, measured on an electropolished surface.
similarities. The responses for both the narrow and the broad specimens are equal within their statistical variation. This is consistent with a processing induced size effect caused by (edge) grinding.

The influence of grinding is further substantiated by grinding a specimen’s surface and performing nano-indentation experiments on the same surface before and after the surface grinding (with indent positions far from specimen edges). In spite of some obvious differences in manipulation between surface and edge grinding, qualitative conclusions can be drawn. Upon surface grinding, the affected surface itself is indented, and not the neighbouring perpendicular surface (as done in edge grinding). Fig. 9 shows the average indentation force at several indentation depths. The solid lines show a series of indentations on a pristine and ground surface. Surface grinding results in an increase in indentation force of about 30% and 16% compared to the pristine state at indentation depths of 1000 and 2750 nm, respectively.

In summary, extensive grinding can be used to remove the edge effect from cutting, as at least the entire edge zone is removed. Yet, grinding introduces a new, minor, processing induced size effect.

3.2. Influence of annealing treatments on processing induced size effects

In order to gain more insight in the nature, extent, and physical cause of processing induced size effects, the optional removal of the edge zone, caused by either of the three manufacturing techniques, is investigated in the following three ways: (1) through additional annealing treatments, (2) moderate grinding of the specimen edges and additional annealing treatments, and (3) extensive grinding and additional annealing.

Option (1) is evaluated for the laser-cut specimens (un-ground) annealed for 1 h at 200 °C (set L_B), optionally followed by an annealing treatment for 0.5 h at 600 °C (set L_C, Table 1). The tensile results for the 200 °C annealed specimens are shown in Fig. 10a. Again, the strength in the low-strain regime (<0.03) significantly increases with decreasing width. The processing induced size effect remains present, in spite of the annealing treatment which only slightly decreased the effect. The degree of recovery is dependent on the type of material and the degree of the induced deformation (Cahn, 1965). Therefore, it is well possible that the above treatment was not sufficient to eliminate the hardening introduced by the processing, i.e. recovery was not complete. To induce further recovery, the specimens have been given an additional annealing of 0.5 h at 600 °C (set L_C), which is 60 °C below the melting temperature of aluminium. This anneal is equal to the recrystallisation treatment used to obtain the base material, see Section 2. The tensile results for this specimen

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3This option allows to make a better comparison of the mechanically cut specimens with the laser-cut specimens, i.e. 1 mm of each of the edge zones of the mechanically cut specimens was removed by grinding (Table 2), leaving an edge zone width approximately equal to the laser-cut specimens.

Fig. 10. Stress–strain curves of laser-cut specimens (a) annealed for 1 h at 200 °C (set L_B) and (b) additionally annealed for 0.5 h at 600 °C (set L_C).
set is shown in Fig. 10b and yield stresses are listed in Table 4. These extensively annealed specimens show a significant drop in their stress–strain response compared to the 200 °C annealed specimens (set LB). Although the statistical variation in the responses is larger, the ‘average’ yield responses of the 2.1 and 4.1 mm wide specimens remain slightly higher compared to the broader specimens, i.e. a small, yet-non-negligible processing induced size effect persists. In order to assess a possibly interfering influence of annealing, the grain sizes and texture of the specimens are next investigated. The grain structure for a laser-cut specimen, in as-prepared condition, after 1 h at 200 °C and after an additional anneal of 0.5 h at 600 °C is shown in Fig. 11. The first anneal (1 h at 200 °C) did not affect the grain structure and texture of the specimen. The second anneal (600 °C), induces some small changes in the grain structure, yet too small to justify the large difference in mechanical behaviour (compare Fig. 3 with 10; see also Table 4).

The presence of an edge zone after an additional anneal of 0.5 h at 600 °C has also been measured by nanoindentation experiments on a 4.1 mm wide specimen of set LC. The results, presented in Fig. 12, show that an edge zone still exists, which is consistent with the observed tensile properties.

Option (2) has been applied to mechanically cut and moderate edge ground specimens, accompanied by an anneal of 1 h at 200 °C (set MB) optionally followed by an anneal of 20 h at 200 °C (set MC) or 8 h at 600 °C (set MD). The results are presented in Fig. 13 and Table 4. The changes in the tensile responses of the various annealed specimens sets look similar to those observed for the annealed laser-cut specimens, see Fig. 10. The yield stress of narrow and broad specimens decreases with increasing annealing time and temperature, an effect which is most profound for the narrow specimens. However, even the extensive anneal of 8 h at 600 °C is not able to completely remove the processing induced size effect, i.e. a significant difference in yield stress remains (Table 4).

Option (3) is used for the specimen ground on the surface, the results are shown in Fig. 9. The anneal for 1 h at 200 °C results in a small decrease of the indentation force, but the force level is still significantly higher than before grinding. Only after an additional annealing step of 8 h at 600 °C, the indentation forces are equal to those of the pristine surface, within their statistical variation. This shows that the processing induced size effect due to the grinding has been reduced to a non-significant contribution (if not completely removed).

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4This specimen is chosen because a possible influence of pre-cutting, which is necessary to prepare the extensively edge ground specimens (sets G), can be ruled out.
Fig. 12. Averaged indentation force at different indentation depths for a 4.1 mm wide laser-cut specimen after 1 h at 200 °C + 0.5 h at 600 °C (set L_C). The vertical bold lines represent the edges of the specimens.

Fig. 13. Stress–strain curves of the mechanically cut and moderate ground specimens (a) annealed for 1 h at 200 °C (set M_B), (b) additionally annealed for 20 h at 200 °C (set M_C), or (c) additionally annealed for 8 h at 600 °C (set M_D).
3.3. Analysis of edge and bulk region properties

To acquire a predictive insight in the strength of the edge region and the processing induced size effect, the average properties of the edge and bulk region are analysed next on the basis of a simple Taylor-type averaging model:

\[
\sigma_u(\varepsilon) = (1 - \eta_w)\sigma_B(\varepsilon) + \eta_w \sigma_E(\varepsilon),
\]

\[
\eta_w = 2 \times \frac{A}{w},
\]

where \(\sigma_u(\varepsilon)\) is the flow stress at a certain strain \(\varepsilon\) for a specimen of width \(w\), \(\sigma_B(\varepsilon)\) and \(\sigma_E(\varepsilon)\) is the flow stress for the ‘bulk’ region and edge region, respectively, and \(\eta_w\) the volume fraction of the edge is dependent on the edge zone width \(A\) and the specimen width. Using Eqs. (1) and (2), the distinct mechanical behaviour of the edge and bulk zones can be extracted from the mechanical behaviour of two specimens with identical edge zones but a different width. The ‘bulk’ behaviour can be determined independent of the edge zone width, whereas for the determination of the ‘edge’ behaviour \(\eta_w\) needs to be known.

The Taylor averaging model was validated with a straightforward finite element (FEM) analysis in which the global uniaxial tensile responses of two specimens with the same edge width, but with different total widths were first simulated. Departing from these global responses, the Taylor averaging model accurately reproduced the individual mechanical behaviour of the edge and bulk zone that was used as input for the simulations. Note that the Taylor model does not take residual stresses into account. Residual stresses are not the cause of the observed processing induced size effects, as was demonstrated by independent X-ray diffraction measurements (not further discussed). The mechanically cut specimens (sets M) are not well suited for the Taylor model analysis, because no bulk zone is present in the 2 mm wide specimens (Table 2), whereas the responses of the ~9 mm specimens alone do not allow to obtain a unique solution. Additionally, it will be shown below that the mechanical cutting to different width also affects the bulk of the material to a different extent. For the specimens of set GB, the edge region introduced by laser-cutting was not completely removed by the extensive grinding. As a result, these specimens are also not well suited for the current analysis. The laser-cut results will therefore be analysed (sets L). The mechanical behaviour of the edge and bulk regions are calculated from the (average) responses of the 4.1 and 9.6 mm wide specimens, with \(A = 1.3\) mm (see Section 3.1.1).

The edge zone width, \(A\), is assumed to be constant, independent of the annealing treatments. The mechanical behaviour of the bulk zone \((\sigma_B(\varepsilon))\) is taken equal for the considered specimens, implying the following assumptions; (i) the bulk material of the specimens is (still) in its recrystallised state, i.e. the material is unaffected by subsequent anneals (supported by Fig. 11) and (ii) the size effect due to lack of statistical microstructural averaging\(^5\) is neglected. The assumption of a constant bulk zone implies that a master curve of the yield behaviour of the bulk region may be determined, which can be used to calculate the edge region properties can be calculated for the various laser-cut specimens.

Using the Taylor model, the bulk curves for the laser-cut specimens in as-cut condition (set LA), after 1 h at 200 °C (set LB), and after an additional 0.5 h at 600 °C (set LC), have been calculated and are shown in Fig. 14a. The bulk master curve is constructed as the average of these three individual bulk curves. This bulk master curve is shown on top of the individual bulk curves in Fig. 14a. The single master curve adequately predicts the response of the bulk region, in particular considering the statistical variations present and the simplicity of the model used.

The yield behaviour of the edge region, \(\sigma_E(\varepsilon)\), in a laser-cut specimen is now calculated using the bulk master curve \((\sigma_B(\varepsilon))\) and Eqs. (1) and (2) with \(A = 1.3\) mm. This is done for both specimens widths (4.1 and 9.6 mm) and for the three different sets. The calculated edge region yield responses in the edge regions are presented in Fig. 14b. In addition, the tensile responses of the 2.1 mm wide specimens are also shown, for which \(A\) is taken equal to half of the specimen width (i.e. no bulk region). Considering \(\sigma_E(\varepsilon)\) for each specimen set (as-cut, 200 or 600 °C), it is clear that the edge responses of the three different specimens widths for each set are in

\(^5\) It is noted that the bulk strength might decrease slightly as the width of the bulk zone decreases, i.e. decreasing specimen width, due to the limited averaging of the bulk crystals present in the specimens (Henning and Vehoff, 2007).
adequate agreement (within the spread of results). Therefore, using a simple Taylor model, the mechanical responses can be predicted, in good agreement, for all specimens of different widths (within a specific set) using a single constant $s_{B}$ and $s_{E}$ with only one degree of freedom being the bulk region width. Comparing the edge and bulk zone properties in the as-cut condition, it is noticed that the yield stress of the edge zone increased up to 250% of the bulk value.

The additional anneals gradually recover the mechanical properties $\sigma_{E}(\varepsilon)$ of the edge regions towards the bulk response (master curve). However, even after a 600 °C anneal the response of the edge region remains considerably higher compared to the bulk. The gradual decrease of the edge zone strength due to annealing suggests that the observed processing induced size effect is caused by dislocations that are trapped in the lattice. This appears to be in contradiction with certain papers in the literature (Humphreys, 1991; Bradley et al., 1976; Perryman, 1954), stating that dislocations run out of aluminium even at room temperature. Specimen processing may induce a local curvature of the crystal lattice in the edge region, which must be accommodated by GNDs. These GNDs are harder to remove from the lattice during recovery annealing, since the overall GND population has to accommodate the existing (non-vanishing) lattice curvature. As a result, the edge zone strengthening cannot be completely eliminated by applying recovery. This observation is supported by X-ray diffraction measurements, which revealed an increased dislocation density towards the specimen edges. Statistically stored dislocations may develop during processing, but they largely run out of the specimen before testing, as emphasised in the literature (Humphreys, 1991; Bradley et al., 1976; Perryman, 1954), where it has been shown that dislocations in aluminium even escape at room temperature. The edge and bulk region properties have also been evaluated for the extensively ground specimens of set GA, which all were mechanically pre-cut to 14 mm. The yield stress of the edge zone, $\Delta = 300 \mu m$, increased up to about 210% of the bulk value.

The yield stress for the bulk region is $\sim 7$ MPa, which is about 25% higher compared to the laser-cut specimens, see Fig. 15. This suggests that the mechanical cutting process also affects the bulk properties of the specimens. The increased yield stress in the bulk is caused by the use of a cushion, applied to the bottom of the specimen during the cutting process, in order to prevent curling of the specimen due to the cutting process. This implies that the extent to which the bulk properties are affected increases with decreasing specimen width.

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6A lower bound for the edge zone width can be determined from the Taylor model itself, i.e. when the edge zone width is taken too small then the Taylor model yields an edge zone material behaviour, $\sigma_{E}(\varepsilon)$, that shows a non-physical softening behaviour. The lower bound corresponds to the smallest value of $\Delta$ for which $\sigma_{E}(\varepsilon)$ shows no softening. This method is used because the nano-indentation results could not be used to determine an edge zone width.
Unfortunately, the nano-indentation results could not be used in this context, since random changes in absolute indentation forces due to specimen mounting, laboratory temperature shifts, etc, prevented a quantitative inter-specimen comparison of the absolute indentation force. To give an indication of the increase in strength in the edges induced by mechanical cutting, the responses of the ~9 mm specimens of set MA ($D = 2\,\text{mm}$) and the bulk curve determined for set GA are used. This results in a yield stress of the edge region, induced by mechanical cutting, that is about 350% of that of the bulk. This is considerably higher than for the case of laser-cutting or extensive grinding, as could already be expected from the comparison of the nature of the three processing techniques.

Finally, a quantitative comparison is made between the differences in yield stress and indentation force between the edge and bulk region, using the data from the laser-cut specimens. The nano-indentation results (forces) are not used to make an estimate of the yield stress (via the hardness), since the commonly used underlying polycrystalline material models are not applicable to the currently used material, where only a few grains are involved in the measurement. In Fig. 14, it is shown that the yield stress of the edge region increased up to 250% of that of the bulk material. This is not immediately obvious from the indentation measurements, see Fig. 5, where the indentation force for the edge region of a 9.6 mm specimen, at about $200\,\mu\text{m}$ from the edge, is only about 14% higher than the force in the bulk region. This results from the fact that only a small fraction of the plastic zone under the indenter is in the low-strain regime. To verify the consistency between the indentation results and the tensile results, simple FEM simulations of the indentation experiment have been performed considering a standard elasto-plastic material using the calculated tensile properties of the edge zone or those of the bulk. Because an axisymmetric FEM model is used, the Berkovich tip was modelled by a conical tip with a half angle of $70.3^\circ$, to ensure that the contact area is the same as that of a Berkovich tip for corresponding indentation depths. In this analysis, contact friction is not taken into account. The FEM analysis predicts an increase in indentation force of about 28% for the edge type material compared to the bulk material. This difference in indentation force between the edge and bulk of about 28% from the FEM simulations is close to the 14% found experimentally. The remaining difference can be explained by the simplicity of the FEM model used or by the fact that the nano-indentation only probes the specimen’s surface region for which the dislocation density may well be significantly smaller than for the specimen interior, whereas the Taylor model yields the material response of the edge zone averaged over the complete thickness.
4. Conclusions

The mechanical behaviour of thin Al sheet specimens with through-thickness grains has been assessed under uniaxial tension. Specimens were prepared from recrystallised plates, assuring a nearly ‘constant’ microstructure. Three types of specimen processing techniques, i.e. laser-cutting, mechanical cutting, and extensive grinding, were applied to make specimens with different widths. From the analysis conducted the following conclusions and original findings are highlighted:

- It is well known that machining induces damage to crystals just below a newly created surface, the effect of which can be safely neglected in macroscopic applications. However, upon miniaturisation, the affected volume increases relative to the geometrical size of the part or specimen. This work has clearly demonstrated that such top-down manufacturing techniques of components and parts introduces a pronounced processing induced size effect in plastic yielding.
- The processing induced size effect upon miniaturisation may cause a significant increase of the engineering yield stress of up to 200% for a decrease from about 12 to 3 grains over the specimen width. The extent of this effect is dependent on the processing methods and the considered material.
- An analysis with a simple Taylor model shows that the average yield stress of the edge region of the cut specimens increased up to 210–350% of the bulk zone value.
- The processing induced size effect is not limited to the specific material investigated here, since it was also identified for commercially available Al sheets with as-received properties (grain size, etc.).
- The processing induced size effect as introduced by the two cutting techniques cannot be completely removed by a subsequent anneal at 600 °C.
- Grinding can be used to remove the edge zones as introduced by cutting, but it introduces a new, yet smaller, processing induced size effect. In contrast to the cutting induced effect, the grinding induced effect can be reduced to a non-significant contribution (if not completely removed) by a prolonged anneal at 600 °C.
- In experimentally driven scientific analysis of size effects, specimens should be prepared with great care, to exclude processing induced size effects, as these may interfere the measurement results. Since processing induced effects are dominantly present in the neighbourhood of machined edges, some tests (torsion, bending) may be even more sensitive to their presence than the tensile case treated here.
- From an industrial point of view, the existence of a processing induced size effect can be incorporated in a predictive design. This work has demonstrated how a simple Taylor model can be adopted to determine the average strength of the edge zone, which can be used to predict the tensile properties of specimens with different geometry. The resulting increase of the yield stress, and the corresponding extension of the plastic regime, are certainly of practical interest.

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