Investigation of the Influence by Size Effects on the Material
Characterization of the Uniaxial Compression Stress State

Submitted: 2021-12-07

Revised: 2022-01-24

Accepted: 2022-02-04

Peter Hetz^{1,a*}, Martin Kraus^{1,b} and Marion Merklein^{1,c}

¹Institute of Manufacturing Technology, Friedrich-Alexander-Universität Erlangen-Nürnberg, Egerlandstraße 13, 91058 Erlangen, Germany

> ^apeter.hetz@fau.de; ^bmartin.kraus@fau.de; ^cmarion.merklein@fau.de *corresponding author

Keywords: Material characterization, Compression test, Size effects, Miniaturization

Abstract. For the evaluation of final component properties under consideration of productioninduced material characteristics, it is necessary to determine the mechanical parameters of parts and structures. Thus, there is a need of new test methods for the material characterization and quality control of produced goods. The miniaturization of the test specimens offers the potential for the local testing of the material properties at almost any position on the component. However, due to the reduction in size of the test specimens, size effects can occur which may affect the material behavior. For this reason, this contribution analyses the influence of size effects on the derived material parameters in dependence of the specimen size and material by using upsetting tests according to DIN 50106:2016 standard. The materials Cu-OFE (copper), X5CrNi18-10 (stainless steel) and AA7075-T6 (aluminum) are investigated to ensure a broad transferability of the findings. The specimens are cut from a bar stock by using a high precision 3D micro electrical discharge milling machining. Due to the small size, every specimen is measured with an optical 3D coordinate measuring system to determine the exact size. Through these investigations, the reproducibility and scatter in the determination of material properties for scaled cylindrical upsetting specimens are evaluated. Furthermore, limits of geometric dimensions at constant aspect ratios are derived. The results of this investigation enable an estimation of the geometrical influence of the specimen size in regard to the mechanical properties.

Introduction

Production companies are constantly challenged to manufacture high-quality products that meet the steadily increasing demands of customers and government institutions. This applies in particular to the area of forming technology, which is dependent on developments in the automotive and aerospace industries. Due to the fundamental structural change towards e-mobility and lightweight construction, the entire industry is facing major challenges. Therefore, it is even more important that quality results are reproducible throughout the entire value chain even in spite of batch and process influences. To meet these challenges, solutions are needed that allow the growing complexity along the process chain to be managed and product quality to be quantified.

In forming technology, material properties are changed during the manufacturing process. Because of dislocation movements, the strength of sheet materials increases with higher degree of forming [1]. In contrast, the plastic deformability decreases. Therefore, it is necessary to observe not only the process parameters but also the material properties of the semi-finished product after a forming process. For this reason, material characterization methods are used not only to numerically map material behavior for simulations, but also for quality assurance and to determine the manufacturing-induced change in component properties. For example, in sheet metal forming flat tensile specimens are used to investigate the mechanical properties in the uniaxial tensile test according to DIN EN ISO 6892-1 [2]. Upset specimens can also be applied to characterize the elastic-plastic material behavior in the uniaxial compression stress state as defined in DIN 50106 [3]. The specimens must be extracted from a plane area with homogeneous material properties in order to be able to determine the characteristics for the respective stress state. To ensure this, the dimensions of the specimens must be

adapted to the geometry of the formed sheet material, e.g., on radii or in the flange, so that they can be extracted. For compression specimens, the minimum realizable dimension of the specimen geometry is limited by the upsetting ratio and the thickness of the sheet material. The limits of the upsetting ratio with the height h_0 and the diameter d_0 of the cylindrical specimen are defined as follows according to DIN 50106 [3].

$$1 \le \frac{h_0}{d_0} \le 2. \tag{1}$$

The specimen height h₀ corresponds to the sheet thickness, if the compression sample is removed perpendicular to the sheet plane. In this case, the specimen diameter according to equation (1) can be as large as the sheet thickness maximum. For this reason, upsetting samples from a sheet are significantly smaller than conventional compression specimens, especially for ultrafine sheets. According to the current state of research, size effects do occur with miniaturized specimens in micro scale, which must be taken into account in microforming technology. Based on Geiger et al., formed parts or structures with two dimensions in the sub-milimeter range belong to microformed parts [4]. Messner et al. have shown that with a reduction in specimen scale, the level of yield stress decreases [5]. This size effect can be divided into grain size effects and geometry size effects [6]. The underlying surface layer model states that grains at the surface of a specimen are less constrained in their motion than grains in the interior part. As dislocations move through the grain boundaries inside the specimen during forming, the strain hardening of the material at the surface decreases. If the scale of the specimen geometry is reduced, the percentage of surface grains becomes larger. This in turn leads to a lowering of the yield stress level [4]. However, in this study, the size effects were only investigated on copper material. It is therefore not proven whether these findings can be applied to other materials. In addition, new measurement methods for acquiring the specimen geometry have been available in the meantime. In this way, the dimensional accuracy of the specimens can be examined much more precisely.

Therefore, in this contribution, miniaturized compression specimens are manufactured from copper Cu-OFE, stainless steel X5CrNi18-10 and aluminum alloy AA7075-T6 by die-sinking EDM followed by an upsetting process. It is investigated to what extent the size effects due to miniaturization can be reduced by improved specimen production. The resulting knowledge can be used to improve the mapping accuracy for the flow behavior of miniaturized compression specimens. In addition, this information can be applied to derive a limit of the specimen size reduction in which size effects influence the level of yield stress. This approach is also relevant for the characterization of a manufacturing-induced change in the plastic deformation capacity and the material strength of formed or additive manufactured components, since these effects can often only be determined with reduced-size specimens.

Test Materials and Specimen Manufacturing

In order to investigate size effects when testing the uniaxial compression stress state, it is necessary to characterize the elastic-plastic material behavior of cylindrical upset specimens with different geometric dimensions. For this purpose, an initial upsetting specimen geometry is used. Both the height h and the diameter d of the specimen are reduced by the scaling factor λ according to [7]:

$$\mathbf{d} = \lambda \cdot \mathbf{d}_0 \tag{2}$$

$$h = \lambda \cdot h_0 \tag{3}$$

The aspect ratio of the specimen remains constant for all variations. As a result, both stress and strain distribution are invariant of specimen size [7]. According to equation (1), the upsetting ratio must be between 1 and 2. Within this investigation, the midpoint of 1.5 is chosen. According to Messner [8], a high ratio can lead to buckling of the specimen. In contrast, a lower one results in an increased frictional force, which in turn causes inhomogeneous deformation of the specimen during the upsetting process. This means that the uniaxial compression stress state can only be mapped to a

limited extent. The functional relationship can be derived from Siebel's upsetting force formula, according to which the reciprocal of the upsetting ratio directly linearly influences the frictional force [9]. Thereby, the punch force F_z is correlated to the specimen area A, the plastic stress σ_{pl} , the friction coefficient μ , the specimen diameter d and the specimen height h.

$$F_Z = -A \cdot \sigma_{pl} \cdot \left[1 + \frac{1}{3} \cdot \mu \cdot \frac{d}{h} \right]. \tag{4}$$

Furthermore, the objective in this publication is to investigate the influence of size effects with regard to the specimen size. For this purpose, it is necessary to exclude other influencing factors such as different friction coefficients of the used materials in the evaluation. The diameter of the specimen with the initial geometry is set to 5.0 mm. With an upsetting ratio of 1.5, the specimen has a height of 7.5 mm. Furthermore, specimens with a λ of 0.4, 0.2 and 0.1 are tested. In this way, following Geiger et al. [4], the materials are upset both in a macro and a micro forming process, since at $\lambda = 0.1$ the geometric geometry in two dimensions is less than 1.0 mm. As a result of the rolling process, sheet materials exhibit a hardening gradient across the sheet thickness and thus an inhomogeneous grain size structure. To ensure that the microstructure of the specimens is homogeneous, the specimens are extracted therefore from a bar stock. The grain structure of the bar material is shown in Figure 1.

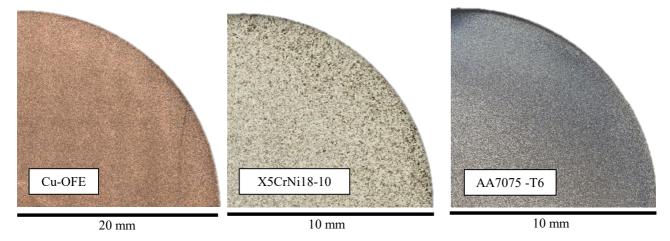


Figure 1 – Etched micrographs of the tested materials over the cross section of the bar material

Except for X5CrNi18-10, the materials show a homogeneous structure over the entire cross section. The stainless steel exhibits a change in the austenite grain structure in the edge region, which is caused by the fabrication process of the bar stock. Thus, no samples are taken from the edge area of the bar stock for all materials in order to ensure a homogeneous grain structure. The sample production procedure is shown in Figure 2. The initial diameter of the bar stock is 40 mm for Cu-OFE. For the other two materials, the bar stock has a diameter of 20 mm. First, slices are separated from the bar material. The height of the slices corresponds in each case to the height of the specimen to be manufactured. Subsequently, the surfaces of the slices are polished, as these are the contact area of the material with the tool in the subsequent upsetting test. In this way, the friction between the compression specimen and the upsetting tool is minimized. Following this, the specimens are separated from the semi-finished product by micro-EDM on an SX-200-HPM (Sarix SA).

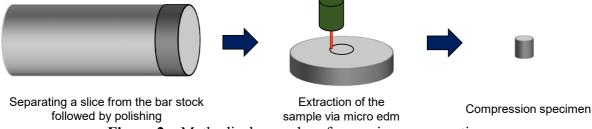


Figure 2 – Methodical procedure for specimen preparation

Usually, semi-finished products are magnetically fixed in position on the EDM machine, but the investigated materials do not have magnetic properties. Hence, they are attached to a magnetic blank by means of a silver-based adhesive. This ensures that the semi-finished product and the specimen are fixed without slipping during the EDM process. After the separation process, the compression specimen is cleaned in an ultrasonic bath to remove the adhesive particles. Before upsetting, the specimens have to be measured. Due to the size of the specimens, it is only possible to measure the geometry by tactile means to a limited extent. For this reason, the optical measuring device InfiniteFocusG5 (Alicona Imaging GmbH) is used. With this system, both the height and the diameter can be determined. Furthermore, roughness peaks and shape accuracy can be detected on a 3D image of the specimen, as shown in Figure 3.

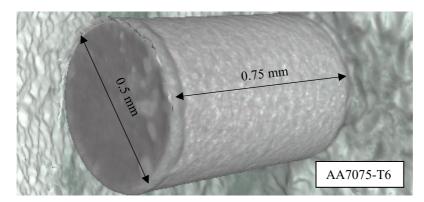


Figure 3 - Optically measured compression specimen ($\lambda = 0.1$)

In order to exclude the possibility that the heat input from the erosion process effects the material properties, micrographs are created of eroded compression specimens made of AA7075-T6 to analyse the grain structure. Especially, Aluminum is susceptible to changes in its grain size and grainstructure as a result of heat treatment. In this way, the edge zones of the sample could soften and lead to falsification of the measurement results. The micrographs are presented in Figure 4. No influence on the grain structure can be detected in the edge zones as a result of the micro-EDM process. Thus, any influence of the erosion process on the material properties can be excluded.

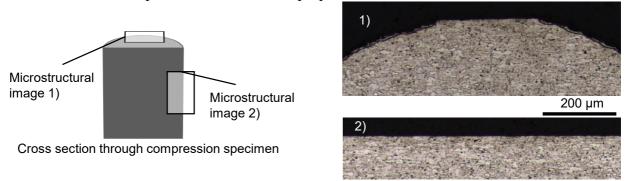


Figure 4 - Grain structure of a compression specimen with material AA7075-T6

Experimental Setup and Evaluation

For the analysis of size effects in the characterization of the flow behavior in the uniaxial compression stress state, upsetting tests with cylindrical compression specimens are carried out. The sample geometries and test materials have been described in section 2. The universal testing machine Z10 (ZwickRoell AG) with a maximum compression force of 10 kN is used. After calibration, a deviation of $\pm 1\%$ of the measured force value was determined around 200 N. Furthermore, due to the low standard deviation of the test results, it can be stated that the load cell measures sufficiently accurately. To ensure that the results are comparable, the test conditions must be identical for all tests. Therefore, the nominal testing speed is 0.0067 1/s and the nominal compression is 50% of the specimen height. Since friction has an influence on the bulging of the specimen and thus the flow

curve is affected [10], the deep drawing oil Dionol ST V 1725-2 (MKU-Chemie GmbH) is applied between the material and the upsetting tools. Thus, it is assured that the friction conditions are comparable throughout all experiments. As shown in Fig. 5, a spherical cap is mounted in the bottom part of the upsetting tool, allowing flexible alignment of the lower tool to the upper tool. The parallel guidance of the tools ensures that the specimen aligns perpendicular to the tool surface during the process despite the high lubricant quantity.

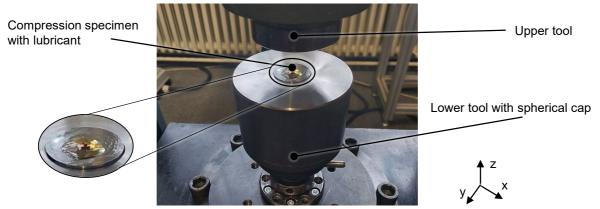


Figure 5 - Experimental setup for compression tests

For this purpose, the fixation of the spherical cap is before testing released and the lower and upper tools are moved together under a compressive force of 1 kN, so that the two tools are oriented parallel to each other. The spherical cap is subsequently adjusted in place so that the tool surfaces are parallel for all experiments and only movement in the z-direction is possible. Both force and displacement data are recorded in z-direction. The displacement data is adjusted with a correction curve that compensates for the stiffness of the universal testing machine and the upsetting tool. The plastic material behavior is determined by reducing the elastic component of the strain using the system module. The strains in x-direction and y-direction are calculated according to the volume constancy under the assumption of an isotropic deformation.

Results and Discussion

In the following, the results from the upsetting tests are presented and discussed. For each material tested (AA7075-T6, Cu-OFE, X5CrNi18-10), the flow curves are compared in a diagram with the respective cylindrical upsetting specimens. All experimental variations were performed with three experimental tests. Subsequently, the flow curves were averaged and plotted with their standard deviation in the diagram. First, the material AA7075-T6 in Figure 6 will be taken into account.

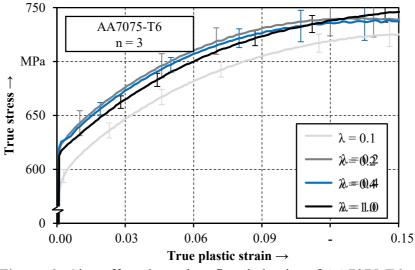


Figure 6 - Size-effect-dependent flow behavior of AA7075-T6

The curves are shown for the aluminum alloy only up to a true plastic strain of 0.15, although the specimens have been upset by 50 %. The reason for this is that the aluminum specimens begin to fail at a strain of 0.15. Thereby the upper side of the specimen shears off from the lower side. As a result, an evaluation for higher degrees of deformation than 0.15 was not possible for this material. In contrast, the materials Cu-OFE and X5CrNi18-10 can be evaluated up to a true plastic strain of 0.5. Within the standard deviation, no size effects can be detected on AA7075-T6 for a λ of 0.2 to 1.0. At a lambda of 0.1, the level of yield stress decreases by about 3 % and so size effects occur. With this the limit for the onset of size effects is between a lambda of 0.1 and 0.2 or respectively within a specimen diameter of 0.5 mm and 1.0 mm. However, the standard deviations are not affected by the size effects, since they are approximately the same magnitude for all variants.

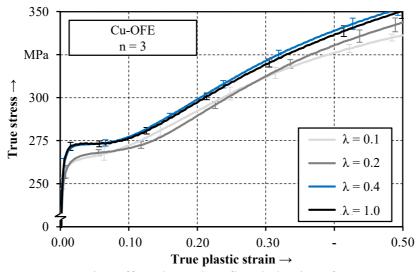


Figure 7 - Size-effect-dependent flow behavior of Cu-OFE

Figure 7 compares the results for Cu-OFE. For this material, size effects appear at a $\lambda=0.2$. The level of the yield stress also declines by approx. 2 %. A further reduction to $\lambda=0.1$ does not lead to a continued decrease in the flow stress. Thereby, for Cu-OFE, the limit for size effects is between a λ of 0.2 and 0.4, whereby $\lambda=0.2$ corresponds to a specimen diameter of 1.0 mm and $\lambda=0.4$ to a diameter of 2.0 mm. The relative standard deviations for this material are about half of that for AA7075-T6. Likewise to Cu-OFE, there is no visible influence of size effects on the standard deviation. Finally, the results for the plastic material behavior in the uniaxial compression stress state for X5CrNi18-10 are presented in Figure 8.

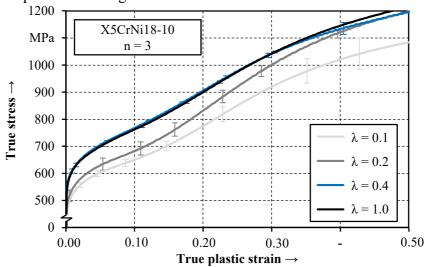


Figure 8 - Size-effect-dependent flow behavior of X5CrNi18-10

In the case of stainless steel, size effects as for Cu-OFE also appear at $\lambda = 0.2$. For $\lambda = 0.1$, the appearance of size effects becomes more pronounced and leads to a reduction of the yield stress level

by about 11 %. Furthermore, with $\lambda = 0.2$, the reduction declines with increasing degree of deformation, so that with a true plastic strain of 0.5, no size effect is apparent within the standard deviation. Furthermore, the material differs from the other two materials in the fact that the standard deviation is influenced by size effects and enlarges considerably from $\lambda = 0.2$. Besides the flow curve, the influence of size effects on the yield strength is also analyzed (Figure 9). The yield strength equates to true stress at a true plastic strain of 0.002 and represents the onset of plastic deformation. It can be observed that for Cu-OFE a reduction of the specimen size has no effect on the yield strength and no size effects occur here. For AA7075-T6, only at $\lambda = 0.1$ is there a slight reduction of 3% in the onset of yielding, which corresponds to the reduction of the yield stress level in the flow curve. In contrast, for the stainless steel, the yield strength is reduced by 13% from $\lambda = 1.0$ to $\lambda = 0.1$.

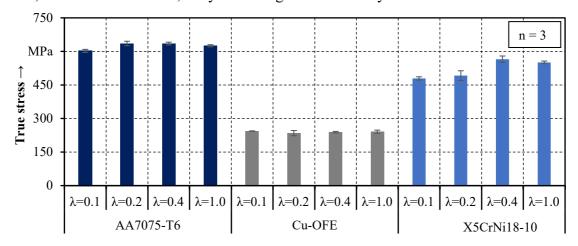


Figure 9 - Size-effect-dependent yield strength

According to the surface layer model the grain size influences the size effect. Therefore, the grain size is investigated using the line intersection method based on DIN EN ISO 643 [11] using the microstructural images of Figure 1. The results are shown in Figure 10. It follows that the grain size increases from AA7075-T6, over Cu-OFE to X5CrNi18-10. Consequently, the grain size correlates with the size effects. Moreover, at a grain size over 20 µm for the material X5CrNi18-10, the flow curve true stress values decrease rapidly compared to the other two materials. However, in order to be able to explain this decline by means of the grain size, further experiments must be carried out with another material and a comparable grain size. Only in this way can the influence of other factors be excluded. Another possible approach is that stainless steel is a multiphase material compared to the other two materials. The phases can also lead to size effects by scaling similar to dislocation movements through the grains.

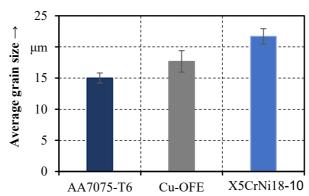


Figure 10 - Average grain size based on the line intersection method in accordance with DIN EN ISO 643 [11]

Conclusion

Based on the results, it can be concluded that size effects have only a minor influence on the flow stress level for aluminum and copper alloys at grain sizes below 20 µm. However, the flow stress level is significantly reduced by 13% for stainless steel. The reduction of the yield stress level correlates with the grain size of the materials. Therefore, for characterization and quality control by miniaturized samples, the grain size has to be taken into account. For this purpose, a limit on the grain size should be defined. Above this limit, size effects occur. The relationship between grain size and the impact of size effects is not linear in this contribution. Further investigations are necessary for the stainless steel material X5CrNi18-10 in combination with a reference material with a comparable grain size. However, it can be concluded that cylindrical upsetting specimens of aluminum up to a diameter of 1.0 mm and copper up to a diameter of 1.5 mm can be characterized at a grain size below 20 µm without size effects.

Acknowledgment

The authors would like to thank the German Research Foundation (DFG) for supporting the present investigations within the scope of the research project "FE-based springback prediction of sheet metal forming processes from lightweight materials considering the anisotropic hardening (DFG project number 403835472)".

References

- [1] D.A. Hughes, N. Hansen, The microstructural origin of work hardening stages, Acta Materialia, 148 (2018) 374–83.
- [2] DIN EN ISO 6892-1: 2017-02, Metallische Werkstoffe Zugversuch Teil 1: Prüfverfahren bei Raumtemperatur, Berlin: Beuth Verlag GmbH.
- [3] DIN 50106: 2016-11, Prüfung metallischer Werkstoffe Druckversuch bei Raumtemperatur, Berlin: Beuth Verlag GmbH.
- [4] M. Geiger, M. Kleiner, R. Eckstein, N. Tiesler, U. Engel, Microforming. CIRP Annals, 50 (2001) 445–62.
- [5] A, Messner, U. Engel, R. Kals, F. Vollertsen, Size effect in the FE-simulation of micro-forming processesm, Journal of Materials Processing Technology 45 (1994) 371–376.
- [6] J.G. Liu, M.W. Fu, W.L. Chan, A constitutive model for modeling of the deformation behavior in microforming with a consideration of grain boundary strengthening, Computational Materials Science, 55 (2012) 85–94.
- [7] U. Engel, E. Egerer, Basic Research on Cold and Warm Forging of Microparts, KEM, 233-236 (2003) 449-456.
- [8] M. Arthur, Kaltmassivumformung metallischer Kleinstteile Werkstoffverhalten, Wirkflächenreibung, Prozeßauslegung, Bamberg: Meisenbach, 1998.
- [9] E. Siebel, Die Formgebung im bildsamen Zustand, Düsseldorf: Stahleisen, 1932.
- [10] M. Lenzen, M. Kraus, M. Merklein, Analytical friction force compensation of flow curves out of layer compression tests with the pin extrusion test, Int J Mater Form, 38 (2020) 405.
- [11] DIN EN ISO 643: 2020-06, Stahl Mikrophotographische Bestimmung der erkennbaren Korngröße, Berlin: Beuth Verlag GmbH.