

Analysis of temperature effect on strength and microstructure in friction induced recycling process (FIRP)

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Keywords: Recycling, Aluminium, Friction-Induced, Energy Efficiency

Abstract. In order to reduce global energy consumption in production and industry along with the associated CO₂ emissions, existing resources must be used more efficiently. This includes the energy-efficient and comprehensive recycling of a wide range of metals. Especially for the production of aluminium, there is a large potential for saving energy using efficient recycling processes. With regard to the recycling of aluminium studies have shown that solid-state recycling processes are significantly more efficient considering the used energy and resources compared to the conventional, smelting-metallurgical recycling process. In this paper, the direct and energy-efficient friction-induced recycling process (FIRP) based on the conform process is further described and analysed in terms of the temperature-property relationships. For this purpose, the influence of the processing temperature on the microstructure and properties of the recycled semi-finished products is investigated using the toll system that enables an ECAP forming. Specific sections of the (in theory) infinite, recycled semi-finished product are taken and analysed at different process temperatures of the solid state recycling process. Based on these sections, the properties in terms of mechanical hardness, strength, ductility and grain size are analysed and a degressive relationship between process temperature and mechanical hardness up to a temperature of 270 °C can be shown. Applying the Hall-Petch relationship, it is analysed whether there is a correlation between the strength and the microstructure in the form of the grain size.

Introduction

In order to achieve the climate targets set in the Federal Republic of Germany for 2030 with regard to the CO₂ emission reduction, emissions must be reduced in all sectors [1]. Aluminium is used as a construction material in a wide application range. Examples of aluminium applications are building facades or the use in the area of vehicles for lightweight construction. In the automotive sector, aluminium is used for lightweight construction to substitute components that are usually made of steel. Due to the low density of aluminium, the mass of the vehicle in operation can be reduced, thus saving emissions. Other properties that further expand the range of applications for aluminium are its good corrosion resistance and the (theoretical) possibility of infinite recycling [2]. However, due to the high emissions, primary aluminium production represents one of the most energy-intensive production processes. The production of one kilogram of aluminium primarily produced from bauxite requires 155.9 MJ of energy. This amount of energy is determined under the assumption of a parity of the underlying energy supply from coal and hydropower plants [3,4]. This value tends to be underestimated due to developments in recent years. Primary aluminium production has shifted significantly in recent years, so that by 2020 most primary aluminium was produced in China [5,6]. At the same time, the share of coal-fired power plants in total electricity generation in China has increased, so that it is more than 60% between 2015 and 2020 [7]. Also not taken into account in such energy balances are various expenses such as the mining and transportation of bauxite as well as aluminium and its precursors. In addition to the high energy consumption, primary aluminium production has a strong impact on the environment and nature due to the mining of bauxite.

One possibility for recycling old as well as new scrap is the secondary production process, which describes the conventional recycling strategy. Heavily contaminated scrap usually has to be freed from oils, greases and other impurities. The processed scrap is then fed into a melting furnace (melting temperature of aluminium is about $T_m = 660$ °C), melted down and subsequently cast into ingots. These ingots are usually processed into profiles or similar using further processes such as an extrusion process [2]. The amount of energy required for the conventional recycling process is significantly lower than the energy required for the primary manufacturing process. Nevertheless, primary aluminium must be included in the production process to some extent, which significantly increases the total energy requirement. If, in addition to the energy required to melt the aluminium, the effort required to dispose of the resulting salt slag is also included in an energy balance, the energy requirement is around 17.7 MJ/kg [8].

Despite the already significantly improved energy balance of the conventionally recycling strategy, there are extensive efforts to further optimize the aluminium recycling process. This is being done both from an energy point of view in the form of a further reduction in the amount of energy used and in an effort to avoid the creation of environmentally harmful salt slag. One way to reduce the amount of energy required is a lower processing temperature in the recycling process [9]. In case the aluminium scrap does not need to be heated to the melting temperature and can be processed at significantly lower temperatures, energy can be saved. Due to this lower processing temperature compared to the melting temperature, solid-state recycling processes are suitable here [10]. An example of such a process is the recycling of pre-compacted aluminium chips using a conventional extrusion process. Due to the fact that large quantities of alloy-pure and uniform aluminium chips are often available from the machining sector, more studies have been conducted in the field of recycling aluminium chips using solid-state recycling processes in recent years. Two advantages of this recycling approach compared to the conventional, melting-metallurgical recycling process of aluminium chips are the significantly lower amount of energy and the saving of material, which is otherwise disposed of in the form of oxides in the salt slag. Furthermore, solid-state recycling processes are usually a direct recycling process. This implies that the produced goods are not in the form of raw shapes such as ingots, but can be directly produced as user-specific geometry such as complex profiles [11]. The basis for achieving good properties comparable to conventionally manufactured products is a major degree of deformation of the parts to be recycled at high temperature at the same time. Only in this way the natural and very hard oxide layer on the aluminium can be destroyed and a high bonding strength between the individual particles be achieved [12]. Friction-induced solid state recycling processes include friction stir extrusion and friction stir consolidation [13]. The processing of aluminium chips with the friction stir extrusion process show that the temperature is a very strong influencing variable of the process forces as well as the properties of the recycled products [14]. Products with a full density and good mechanical properties can be produced, but the discontinuity is a limitation for the process [15]. Another solid-state process for recycling aluminium chips has recently been investigated at the Department of Forming and Machining Technology (LUF) at Paderborn University. Compared to the recycling process based on conventional extrusion, the friction-induced recycling process (FIRP) provides the advantage that profiles can be produced in unlimited lengths. In previous investigations, it was shown that a full profile with good mechanical properties of the wrought alloy EN AW-6060 could be produced in the stationary production phase of the friction-induced recycling process. Hardness measurements show a homogeneous distribution of properties over the entire cross-sectional area. The profiles produced show significant grain refinement within the microstructure compared to reference wires. Also for this recycling process a reduction of energy input could be achieved compared to secondary aluminium production [16,17].

The central motivation for the investigations presented here on the continuous and friction-induced recycling process is the investigation of the process parameters and influencing variables

necessary for solid state recycling. The friction induced recycling process is well suited for these parameter studies due to the steady state nature of the process. Within this stationary state, the key parameters of temperature, torque or extrusion pressure as well as the processed mass flow rate show constant characteristic values. A detailed analysis of the conditions required for the production of profiles is well possible in the steady state of the friction-induced process and is investigated in this publication as part of the determination of the microstructure properties correlation.

Processing Principle and Materials

The machine set up shown in Fig. 1, a) provides the basis for the investigations concerning the friction-induced recycling process described in the following. By using the here presented setup a wide variety of aluminium scrap, in terms of geometric size, can be processed. Therefore the material to be recycled is fed into the groove (6x7 mm) of the rotating wheel at a constant mass flow rate. The wheel is fixed on the output shaft of a powerful geared drive, which rotates at a constant rotational speed in the range between $n = 6 - 11$ rpm for the investigations described in this paper. A tool insert continuously reduces the cross-sectional area of the wheel groove by engaging in the groove of the wheel. This steadily compacts the aluminium chips, which initially have a very low bulk density. While the aluminium particles rotate within the wheel groove, they are compressed and plasticised, so that at the end the density of the aluminium is increased to $\rho = 2.7 \text{ g/cm}^3$. Following the quarter turn, the entire cross-sectional area of the wheel is filled by a counterholder which deflects the material. The additional deformation of the deflection further breaks up the oxide layers of the material. Subsequently, the material is extruded through a shaping die in a manner analogous to extrusion.

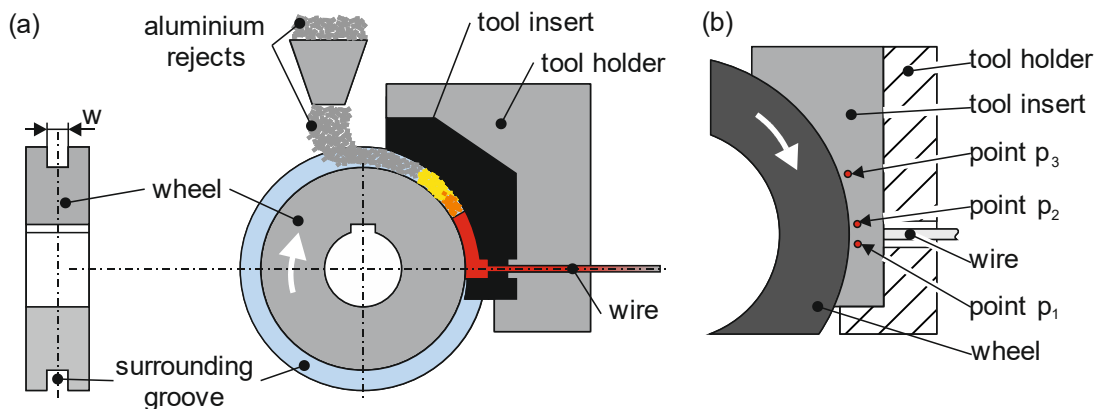


Fig. 1. Set up for the friction-induced recycling process. (a) Tool system. (b) Illustration of the temperature measuring points.

Central indicators of the friction-induced recycling process are the temperature as well as the torque applied by the gear motor (SIMOGEAR parallel shaft gear motor, Siemens AG). The torque is determined from an average value of the electrical power consumption of the frequency converter (SINAMICS G120, Siemens AG). The direct temperature measurement inside the extrusion channel to determine the true material temperature is very difficult. Instead, a tactile measuring principle is used to record the real temperature on the outside of the die insert at different measuring points depicted in Fig. 1, b). Type K thermocouples (G/G-24-KK-IEC, Therma Thermofühler GmbH) and an MX440A measuring amplifier (Hottinger Brüel & Kjaer GmbH) are used for this purpose. In validation tests it has been proven that this type of temperature measurement approximates the actual forming or extrusion temperature well. This could be demonstrated with the measurement of the temperature a few millimetres in front of the extrusion

channel as well as the exit temperature of the recycled wire, which corresponds to the temperature measured on the outside at points p_1 and p_2 in the stationary state.

Irrespective of the possibility of processing a wide variety of aluminium scrap, only aluminium chips are used for the investigations in this paper. The reason for this is the fact that these can be produced specifically under constant cutting conditions. This results in a constant chip size, which is necessary for setting a constant mass flow in the feed and thus also constant test conditions. The cutting conditions used for the production of the aluminium chips of the alloy EN AW-6060 (T66) as well as the resulting chip geometry are given in Table 1.

Table 1. Cutting conditions and resulting geometry of the chips used.

Machining parameters		Chip Size (Material EN AW-6060)	
Cutting speed	$v_c = 1425$ m/min	Raw Material	plate 300x200x20 mm
Feed	$f = 0.2$ mm	Chip length	$l_c \approx 1.9 \pm 0.12$ mm
Cutting depth	$a_p = 2$ mm	Chip width	$w_c \approx 1.5 \pm 0.1$ mm
Cutting width	$a_e = 0.5$ mm	Chip thickness	$t_c \approx 0.095 \pm 0.007$ mm

Results and Discussion

For a wide industrial application, the productivity of a process is an important decision parameter for the operational application in addition to the economic efficiency. The rotational speed of $n = 6$ rpm used in previous investigations results in a lower discharge and heating rate compared to conventional extrusion. This is to be improved in a first step by increasing the wheel rotational speed to $n = 11$ rpm in combination with a proportional increase in the filling rate. With the relationship described above, the discharge rate can be almost doubled. Fig. 2 shows the measured values of temperature and torque recorded in a test with $n = 11$ rpm. The torque is calculated from the electrical power consumption of the frequency inverter and represents an average value due to natural fluctuations. The three measuring points relevant for the temperature measurement are shown in Fig. 1, b) above. Points p_1 and p_2 are located below and above the extrusion channel, respectively. Measurement point p_3 is located further up on the tool, which can be seen as an explanation for the consistently lower temperatures compared to the other two measurement points.

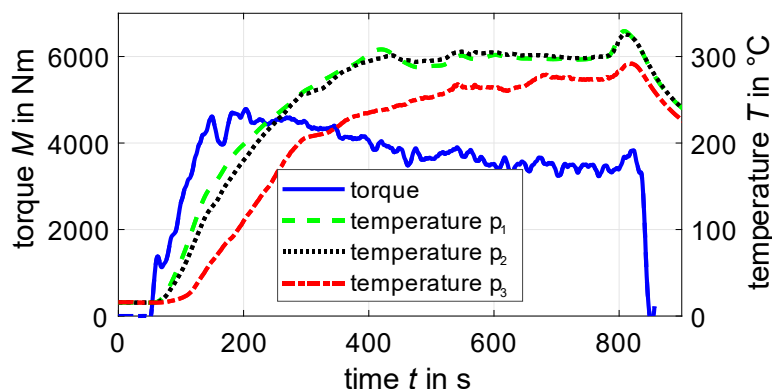


Fig. 2. Torque and tool/process temperatures for different measuring points.

At the beginning of the process (after approx. 45 seconds), the torque rises sharply when the gear motor is switched on and the chips are directly fed in, and initially remains at a high plateau. Analogous to the torque, the temperature readings also initially show a sharp increase. After a total time of approx. 435 seconds, a steady state is reached at nearly $T = 300$ °C with respect to the temperature values of measuring points p_1 and p_2 . From the time the stationary process state is

reached the temperatures of these measuring points remain constant except for a slight increase when chip filling stops at the end of the process. With a delay of approx. 160 seconds, the torque also reaches a stationary state at $M = 3450$ Nm. In a direct comparison of the results presented here for the high rotational speed of $n = 11$ rpm compared to the previously published results with $n = 6$ rpm, there is a clear increase in the heating rate, so that the steady state is reached significantly earlier.

Temperature property correlation.

The aim of the investigations presented here is to describe the relationship between the mechanical strength and the temperature that prevails in the process. Due to the homogeneous temperature distribution within the die insert, the process temperature is equated with the temperature measured at the first or second point on the outside. To illustrate the influence of different rotational speeds ($n = 6, 8, 10, 11$ rpm) on the temperature balance as well as the mechanical properties, investigations and measurements have been carried out at different rotational speeds which shows macroscopic good properties of the recycled wires. For all four rotational speeds investigated, the process temperature is assigned to a specific location on the extruded wire by means of a time-dependent marker. This enables a precise analysis of the individual test points after the extrusion process to reveal the correlations between rotational speed, temperature and mechanical properties. The temperature was determined for all three rotational speeds at point p₁. Depending on the rotational speed and thus also the discharge rate, between 6 and 22 test points were recorded for each rotational speed. Even in the instationary process state for low temperatures < 200 °C, a homogeneous wire is produced with only minor inhomogeneities on the surface (see Fig. 3, a). Following the test, the wires were cut at the previously defined test points, embedded and subsequently subjected to a hardness test. The measured value of each test point represents the average of five individual measurements on the head surface (see Fig. 3, a) to counteract and compensate any uncertainties. Fig. 3, b shows these average values for the test points of all four rotational speeds (marked in colour) along with the temperature assigned to the respective point. For all four rotational speeds considered, the hardness decreases in the instationary start up phase as the temperature increases until the temperature of 270 °C is reached. In general, the original strength of the starting material is significantly reduced.

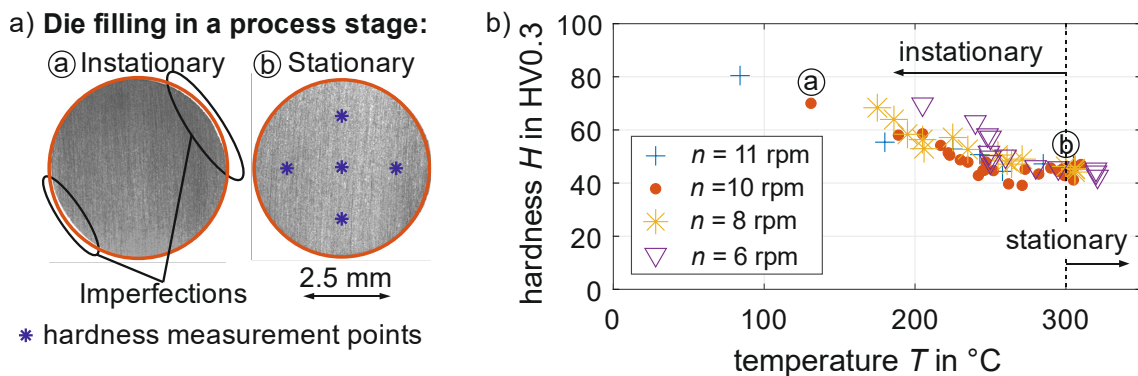


Fig. 3. Temperature-dependent development in, a) die filling, b) hardness as a function of temperature T and rotational speed n .

The mechanical hardness of the tubes used to machine the chips is about $H = 90$ HV 0.3 (in a T66 condition). The application of temperature in the extrusion process significantly reduces the strength or measured hardness due to recrystallisation. This reduction in the initial strength of the material due to the introduction of temperature has also been observed in comparable processes such as friction spinning. In the context of friction spinning, which also obtains the temperature from the friction between the forming tool and the workpiece, similar hardness values of between $H = 40 - 50$ HV 1 are obtained following the process [18].

Considering the temperature range above 270°C, which represents the steady state, it first becomes apparent that, due to the only slight temperature change, many measuring points show almost identical hardness values. This confirms the previously made assumption of constant properties within the steady state. Due to the fact that the stationary process state has been reached, no further heating will take place in the natural process. Regarding the influence of the rotational speed on the hardness, it can be summarised that in the stationary state of the temperature range considered the hardness is not influenced by the rotational speed. A check of the behaviour for temperatures above 310°C measured on the outside of the die must therefore be carried out in future investigations at higher rotational speeds, using modified tools or with the aid of heating cartridges.

One requirement for the use of products made from recycled materials is a consistent and reliable workpiece quality. In the friction-induced recycling process which is investigated in this paper, a steady state is established when a defined temperature level (for the above described trial with $n = 11$ rpm the stationary temperature is nearly 300°C) is reached. In the following, the extent to which the workpiece quality is consistent from the time the steady state is reached is analysed. The hardness distribution on the cross-sectional area of wire sections in the steady state of the friction-induced extrusion process of the high rotational speed of $n = 11$ rpm is analysed. Six wire sections from $l = 2.5$ m to $l = 5$ m were tested. It can be shown that the hardness values on the head surface vary only slightly around the respective mean value of the section. The standard deviation of the respective sections must therefore also be compared to the reference wire. The mean value of all 78 measurement points is $H = 47.8$ HV 0.3 with a standard deviation of $SD = 2$ HV 0.3. The consideration of the results of tensile tests also show a uniform average tensile strength of $R_m = 155$ MPa with a standard deviation of $SD = 4.5$ MPa. From these measurements, it can be concluded that friction-induced extrusion can be used to produce semi-finished products within the steady state that exhibit a homogeneous distribution of the mechanical property of hardness and tensile strength. The hardness and tensile strength of the recycled samples are comparable to the properties of semi-finished aluminium products of the alloy EN AW-6060 in the solution-annealed and softened T4 condition according to EN 755-2:2016. The softening can be attributed to the dynamic recovery and recrystallisation caused by temperature and deformation.

Microstructure Strength Correlation.

The decreasing strength of the recycled wires with increasing temperature will be explained in the following based on an analysis of the microstructure (grain size) on the cross-sectional and the longitudinal-sectional area surface of the wires. For this purpose, four sections produced at different temperatures, at the rotational speed of $n = 10$ rpm are embedded and electropolished for each area. The aim of this is to observe the microstructure and analyse the grain size. Fig. 4 shows the development of the microstructure and thus also of the grain size for four different test points, each at a different temperature.

The first test points (I) were generated at a temperature of $T_1 \approx 247^\circ\text{C}$ and shows small, uniformly distributed grains with an average grain size of $d_{k,1} \approx 3.5$ μm on the cross-sectional and $d_{k,1} \approx 3.9$ μm on the longitudinal-sectional area. For the second as well as the third test point, the grain size increases with the temperature. This is valid for both the cross-sectional and the longitudinal-sectional area independent of smaller deviations between the two different areas. For the highest temperature considered of $T_4 \approx 310^\circ\text{C}$, an average grain size of $d_{k,4} \approx 6,3$ μm on the cross-sectional and $d_{k,4} \approx 6,4$ μm on the longitudinal-sectional area can be measured. From these results, a clear progressive relationship between temperature and grain size can be determined.

The reduced hardness and growing grain size with increasing process temperature can be described by the microstructure restructuring due to recrystallisation in agreement with the Hall Petch relationship. In general, a dependence of the mechanical properties in terms of a hardening or softening can be made as a function on the grain size. This relationship is described by the Hall-

Petch equation. The yield strength or 0.2% yield point σ_y is determined on the basis of the material constant σ_0 which take the required stress for dislocation movement into account and a constant of yielding K as well as the average grain size d according to Eq. 1 [19,20].

$$\sigma_y = \sigma_0 + K \cdot d^{-1/2} \tag{1}$$

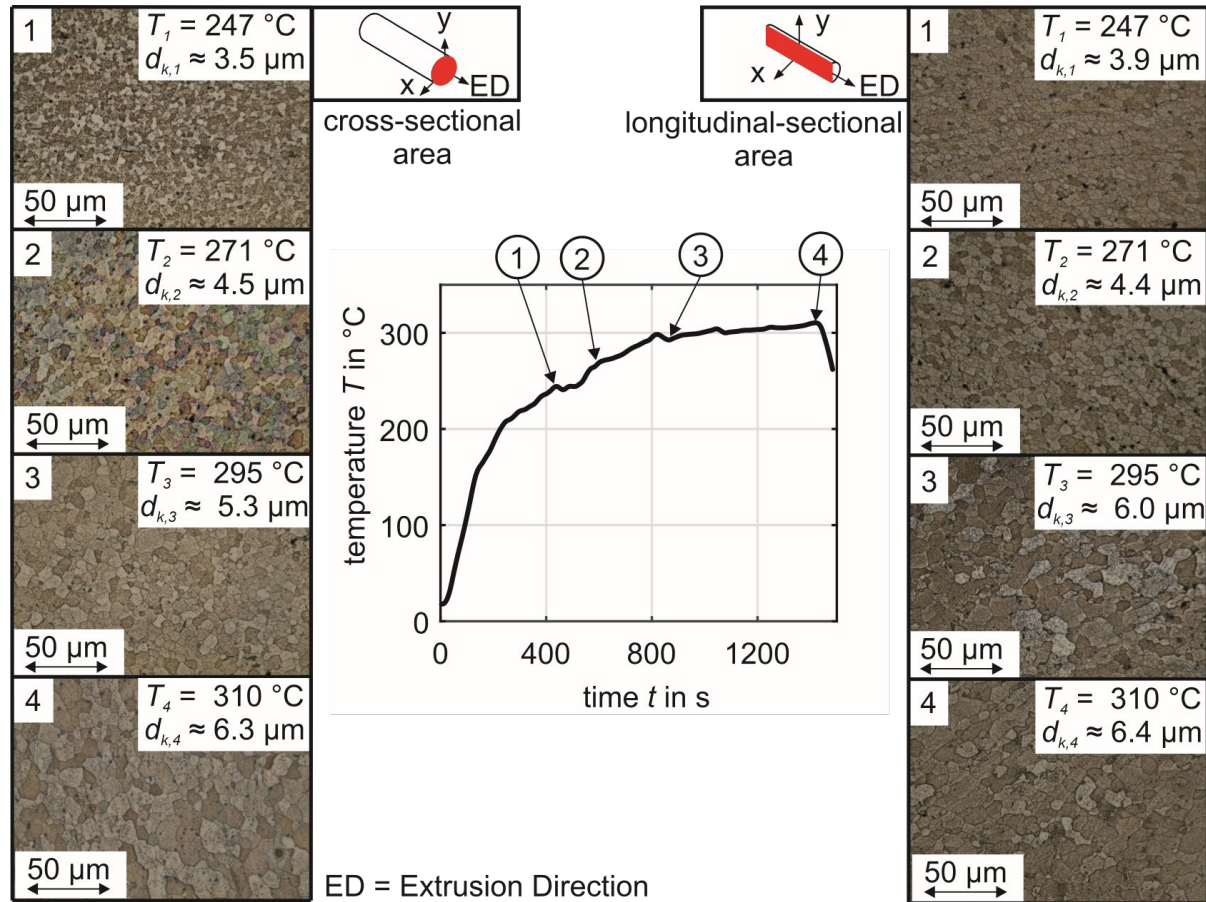


Fig. 4. Change in microstructure of wires produced at 10 rpm as a result of temperature changes measured on the outside of the tool.

As previously described, the grain size changes as a function of temperature in the friction-induced recycling process for both considered intersections. In order to verify the validity of the Hall-Petch relationship for this process, tensile tests of the individual sections are carried out. The results of the tensile tests in comparison to the temperature and the grain size are show in Table 2.

Table 2. Change in temperature, grain size, and yield strength.

Point of investigation	Temperature T in $^{\circ}\text{C}$	Grain Size cross-sectional area d in μm	Grain Size longitudinal-section area d in μm	yield strength σ_y in MPa
1	247	3.5	3.9	104
2	271	4.5	4.4	98
3	295	5.3	6.0	95
4	310	6.3	6.4	93

Due to the steady increase in temperature, the tensile tests for the four points described above represent a temperature interval of $\pm 5^\circ\text{C}$ around those used to determine the grain size. Each result of the yield strength shown in Figure 6 represents the average of two to four measurements (depending on availability). For the cross-sectional area, the constants are determined to be $\sigma_0 = 59.8 \text{ MPa}$ as well as $K = 82 \text{ MPa } \mu\text{m}^{1/2}$ with a high coefficient of determination of $R^2 = 0.988$, and for the longitudinal-section area, the constants are determined to be $\sigma_0 = 59.5 \text{ MPa}$ as well as $K = 85 \text{ MPa } \mu\text{m}^{1/2}$ with a coefficient of determination of $R^2 = 0.9$. In studies on the microstructural changes of equal channel angular pressing, a constant of $K = 114.3 \text{ MPa } \mu\text{m}^{1/2}$ has been determined for the alloy EN AW-6060, which is also investigated in this paper. For the nearly pure EN AW-1100, a slightly lower constant of $K = 105 \text{ MPa } \mu\text{m}^{1/2}$ is determined [20]. Other sources calculate $K = 65 \text{ MPa } \mu\text{m}^{1/2}$ [21] or $K = 70 \text{ MPa } \mu\text{m}^{1/2}$ [22] for the pure aluminium. The values investigated within the scope of this paper shows certain, but not too large deviations from the literature values. However, this can also be attributed to the smaller number of study points considered ($n = 4$). In general, it can be stated that both the results obtained from the experimental work in terms of yield strength and grain size correlate according to the Hall-Petch relationship and the strength mechanisms can be attributed to the change in grain sizes. A direct comparison of the friction-induced recycling process with similar forming processes for the production of semi-finished products shows that (when using the tool system of the friction-induced recycling process described here) a lower process temperature occurs compared to hot extrusion. This, coupled with the significantly shorter contact time of the process temperature in the recycling process, can be used as an explanation for the significantly smaller grain size of the recycled profiles compared to profiles produced with the hot extrusion process [23,24].

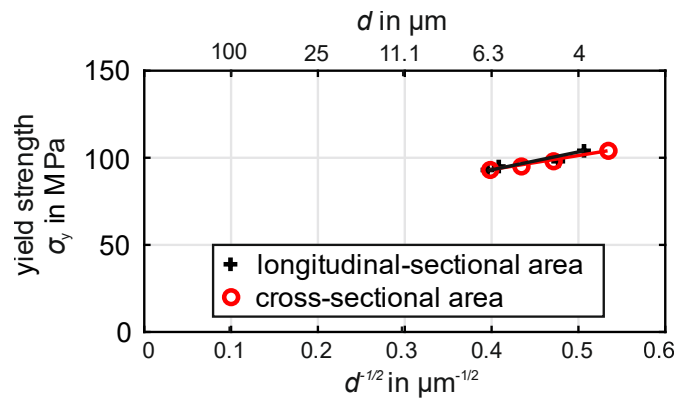


Fig. 6. Dependence of the mechanical strength (yield strength) on the grain size.

Summary

This paper presents further investigations on the energy-efficient and resource-saving friction-induced recycling process. Depending on the tool system used, a steady-state process condition is established after the heating phase, which was demonstrated by constant hardness values in the temperature range above 270°C. Further investigations also show a dependence of the microstructure in the form of the grain size as well as the strength in terms of the yield strength, which are modelled and represented using the Hall-Petch relationship. Considering the best practise in developing a resource and energy efficient recycling process the rotational speed of the wheel does not affect the hardness of the recycled profiles in the stationary production phase. Using higher rotational speeds can be useful to increase the output while recycling particles/ chips with a low density. First fundamental research shows the possibility of raising the processing temperature by increasing the extrusion channel length of the tooling system due to the increase in frictional surface considering the stationary production phase. These and further possibilities (e.g. modification in extrusion ratio) to affect the process temperature and mechanical properties of the products will be the aim of future investigations.

Acknowledgment

This research was funded by the state of Northrhine Westfalia via the Forschungskolleg 'Leicht-Effizient-Mobil' (LEM).

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