Production of ultrafine grained aluminum by cyclic severe plastic deformation at ambient temperature

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Abstract. In the present study the possibilities of grain refinement was investigated by applying large-scale of cyclic plastic deformation to aluminum at ambient temperature. The specimens are processed by multiaxial forging, which is one of the severe plastic deformation techniques. The aim of the experiments with the aluminum alloy 6082M was the determination of the equivalent stress and strain by multiaxial forging and the investigation of evolution of mechanical properties in relation with the accumulated deformation in the specimen. The mechanical properties of raw material was determined by plane strain compression test as well as by hardness measurements. The forming experiments were carried out on Gleeble 3800 physical simulator with MaxStrain System. The mechanical properties of the forged specimens were investigated by micro hardness measurements and tensile tests. A mechanical model, based on the principle of virtual velocities was developed to calculate the flow curves using the measured dimensional changes of the specimen and the measured force. With respect to the evolution of these curves, the cyclic growth of the flow stress can be observed at every characteristic points of the calculated flow curves. In accordance with this tendency, the evolution of the hardness along the middle cross section of the deformed volume has also a non-monotonous characteristic and the magnitudes of these values are much smaller than by the specimen after plane strain compression test. This difference between the flow stresses respect to the monotonic and non-monotonic deformation can be also observed. The formed microstructure, after a 10-passes multiaxial forging process, consists of mainly equiaxial grains in the submicron grain scale.

Keywords. Severe plastic deformation, MaxStrain System, Multiaxial forging, Grain refinement, Flow curves at large plastic strain

1. Introduction
In recent years, bulk ultrafine grained and nanostructured materials are processed by different methods of severe plastic deformation (SPD). Large amounts of plastic deformation can have considerable effects on the microstructure as well as the mechanical and physical properties of metals and alloys. The SPD techniques (equal channel angular pressing, multiaxial forging, high pressure torsion, etc.) can provide very large plastic strain, characterized by shear and so-called non-monotonic deformation. The formed microstructure is granular containing mainly high angle grain boundaries, in contrast to the conventional deformation processes (cold rolling, drawing, etc.) which can result usually in substructures of cellular type having boundaries with low angle misorientations [1,2].

One promising process of SPD methods is the multiaxial forging whereby metals are forged free [3] or in a channel die [4] repeatedly and sequentially in three orthogonal directions at room or elevated temperature. A special experimental setup of multiaxial forging is realized in the MaxStrain System of Gleeble thermo-mechanical simulator. In this process, the specimen is compressed cyclic in two mutually orthogonal directions in parallel with controlled temperature profile [5]. The mechanism of grain refinement during multiaxial forging with MaxStrain System was studied on aluminum [5] or copper [6]. A quantitative model of grain refinement and strain hardening was developed by Petryk et al. [7]. The evolution of mechanical properties was also investigated on HSLA and IF steels [8] as well as iron aluminum alloy [9] deformed at elevated and room temperature using the MaxStrain
Technology. The fracture behavior of different ultrafine grained aluminum alloy, processed by also this method, was studied under static and cyclic loading [10].

In the studies about MaxStrain Technology several investigations are presented as described above, but the accurate knowledge of the states of stresses and strains would be needed. The primary aim of recent multiaxial forging simulations was to develop a mechanical model for direct calculation of flow curves. In the frame of a complex investigation, the evolution of micro hardness, tensile behavior and microstructure were also examined on the series of one to ten times forged specimens.

2. Experimental procedures and modeling

The experiments at room temperature were performed on AlSi1MgMn heat treatable aluminum alloy. The aluminum specimens were cut from extruded raw material followed by annealing at temperature of 380°C for two hours and cooled down to room temperature at the rate of 1°C/min. This treatment stabilized the microstructure in order to avoid the natural aging effect. The mechanical properties were determined by plane strain compression tests using the Watts-Ford method as well as by tensile tests and micro hardness measurements. The multiaxial forging experiments were carried out on Gleeble 3800 thermo-mechanical simulator using MaxStrain System. The experimental setup of multiaxial forging technique (cyclic compression test) is shown in Fig. 1.

![Experimental setup of multiaxial forging simulation](image)

Figure 1. The schematic view (a) and a photo (b) of the experimental setup of multiaxial forging simulation in MaxStrain System.

In this configuration, the simulator provides the possibility of applying controlled deformation schedule even at high temperature on various metals and alloys. Two uniaxial pistons with mounted forming tools are moved by a fully integrated hydraulic system, controlled by high-speed servo valves in an enclosed, digital PID control loop. Different transducers (for example longitudinal and transversal strain gauges, load cell, etc.) generate the feedback to insure the accurate control of hydraulic compression rams. The specimen is positioned in the manipulator by the clamping grooves in order to restrain the axial deformation and rotated between the forging passes back and forth by 90°. The reduced center part of the specimen with dimensions of 12x12x10mm, hereinafter referred to deformed volume, is formed between two tungsten carbide prismatic anvils.

2.1 Multiaxial forging experiments and material testing

Ten simulations were performed such that the number of applied passes was increased one by one per specimen from 1 to 10. This simulation series was repeated on another ten specimen. Before the deformations, the anvils approach and contact the specimen according
to the specified force-criterion then the program calculates the height as well as the width of
the deformed volume – depending on the position of the specimen in the manipulator –
using the initial height or width of the specimen and the actual signal of the LVDT-
transducer. During the deformations, the amount of strain and the strain rate in the direction
of the hydraulic pistons are also controlled by the signals of the longitudinal displacement
transducer [11].

In the tests two deformations are applied in the same direction: a pre- and a main-
deformation with strains of 0.1 and 0.4, at a strain rate of 0.1 1/s in order to obtain a quasi-
prismatic shape before the main-deformation, characterized by measured initial width and
height dimensions. Using the measured and sampled dimensions, the initial and final area of
the contact surfaces between the deformed volume and the anvils can be also calculated. To
expand the investigation, a 40-passes simulation was carried out in order to reach the typical
strains of SPD processes by achieving more than 15 in accumulated equivalent strain.

In transversal direction, the ten specimens were cut in half and polished then the Vickers
hardness was measured on the cross sections by micro hardness testing machine in gridded
layout with a spacing of 0.5mm. On average, about 120-150 hardness values were measured
per specimen. Using the other series of forged specimens, plate samples were manufactured
for tensile tests. Due to the small dimensions of the deformed volume, special specimens with
non-proportional geometry were machined. The gauge length was 6mm and the dimensions
of the cross section were 1.5 to 2mm thickness and about 3mm in width. A specimen after the
10-passes forging simulation was used for preparing foils parallel to the directions of
deformation to investigate the micro- and substructure by Transmission Electron Microscopy
(TEM).

2.2 Mechanical model

The mechanical model of multiaxial forging was developed based on the principle of virtual
velocities. To calculate the actual flow stress $\kappa$, the real velocity field and the external
pressure which acts trough the anvils on the contact surfaces of the deformed volume, has to
be known exactly. Using the dimensional changes of the deformed volume, the real velocity
field can be calculated. The external pressure was obtained from the measured force data and
the calculated area of the contact surfaces. If using the principle of virtual velocities in the
functional of plasticity and it is determined at its stationary point, the actual velocity field is
given. The material is assumed to isotropic and incompressible and the inertia forces are
suppressed [12]. The functional of plasticity is given by

$$J = \int_V \int_{\sigma} dV + \int_{\tau} \tau' \Delta v, dA + \int_{\tau_s} \tau_s \Delta v_s dA - \int_{\tau_s} \tau_s v_s dA - Q' v_s$$

(1)

The first term expresses the internal power over the deformed volume. The second term
covers the frictional power on the contact surfaces $A_v$, the third term defines the shear power
over the surface of velocity discontinuities $A_{\tau}$, connected the deformed volume to the shaft of
the specimen. The fourth term represents the power of the $t'$ stress vectors acting on surface
$A_{\tau}$, which corresponds to $A_v$; this term is zero in this model. The last term includes the
power of force $Q'$ acting on the rigid shaft, which are connected to the deformed volume $V_s$.
The variation of this functional is zero at its stationary point. By the solution of the
mechanical problem the material was assumed as a rigid-plastic and the flow stress is
assumed to constant overall the deformed volume in each moment of time. Based on the
measured dimensional changes, instead of the kinematically admissible velocity fields the
real one can be used, assuming that the width of the specimen changes according to [13]:
\[ W = W_0 \left( \frac{H}{H_0} \right)^{\alpha_0} \]  

(2)

where \( H_0, H \) are the initial and the actual height, \( W_0, W \) are the initial and the actual width of the deformed volume and \( \alpha_0 \) is the variational parameter, respectively.

Arranging and integrating the equations of the velocity field, the variational parameter can be determined from the initial and final dimensions of the deformed volume. The change in the area of the contact surfaces during the deformation passes is described using the evolution of the width at contact surfaces expressed also by the formula of Eq. (2). After the external pressure on the contact surfaces was calculated, the externally supplied power \( (J) \) can be expressed. Finally, if the terms in the functional in Eq. (1) is differentiated by the variational parameter, the following variational function is given, which can be determined based on the Ritz method [14].

\[
\delta \{ \dot{W}_r + \dot{W}_t + \dot{W}_\Delta - \dot{W}_g \} = \frac{d}{da_0} \{ \dot{W}_r + \dot{W}_t + \dot{W}_\Delta - \dot{W}_g \} = \frac{d\dot{W}_r}{da_0} + \frac{d\dot{W}_t}{da_0} + \frac{d\dot{W}_\Delta}{da_0} - \frac{d\dot{W}_g}{da_0} = 0 \]  

(3)

From Eq. (3) the pressure acting on the rigid shaft of the specimen is expressed as a function of flow stress. Producing the quotient of the external pressure and the flow stress, the actual flow stress \( (k_r) \) can be determined from Eq. (1) at a given state.

3. Results and discussion

The sampled force data and the dimensions of deformed volume are plotted in Fig. 2. Owing to the measuring algorithm, the initial and final height as well as the width of the specimen can be determined at every forging pass. Cyclic increase and decrease of these dimensions observed definitely and the difference between theirs values at the odd and even passes is eliminated as the number of forging pass is increasing. Using these dimensions the apparent decrease of the deformed volume was calculated and applied in the mechanical model. Moreover the relation between \( W_{max} \) and \( W_{max} \) is used to determine the friction factor. On the graphs of force versus change of height, the odd and even passes can be also differentiated from each other.

**Figure 2.** The measured force data versus corrected change of height (a) and the dimensions of deformed volume (b) by the 40-passes multiaxial forging simulation.

A mathematical algorithm was written to calculate the actual flow stress. With respect to Eq. (1), the actual value of flow stress was computed using the instantaneous value of force.
and the height and width of the deformed volume as well as the actual area of the contact surfaces. The calculated flow curves of the 40-passes multiaxial forging simulation and the reference flow curve obtained by plane strain compression test are shown in Fig. 3. The results of compression test and the calculated flow curve of first forging pass show approximately the same flow stresses at the common range of the equivalent strain. The first ten curve can be seen on the enlarged graph. As the accumulated strain is increasing, the stress is growing monotonically up to the fourth pass, however at specified equivalent strains the magnitude of these values are smaller than the flow stresses obtained by the plane strain compression test. From the fifth flow curve concerning the odd as well as the even forging passes, the increase is also monotonic, but much less intensive. Comparing the odd and even curves, a cyclic growth can be observed.

![Figure 3](image)

**Figure 3.** The calculated flow curves for the 40-passes multiaxial forging simulations and the enlarged graph of the first ten forging pass.

In Fig. 4 the first and the second flow curve as well as the calculated maximum stresses of odd and even deformation passes are plotted separately. The Voce-type equation was modified and used to fit curves to the presented points of the odd and even forging passes, respectively. There is significant difference between the hardening exponents of these enveloping curves.

![Figure 4](image)

**Figure 4.** The fitted enveloping curves to the flow curve of 1st as well as 2nd pass and to the maximum stresses of odd and even passes, respectively.
The measured hardness values of the ten forged specimens were analyzed separately and the normed relative incidences were constituted and plotted in histograms, as presented in Fig. 5/a. The Weibull-distribution was used to describe the generated incidences. The density function were fitted to these graphs and the three characteristic parameters (α, β and µ) were determined for each distribution. The median, the modus and the deviation was calculated using these parameters. The medians and the average of maximum and minimum hardness values (calculated from the 10% of measured hardness values which are the largest and smallest by each specimen) versus the accumulated equivalent strain by the annealed initial state, by each stage of plane strain compression test and by the ten forged specimens are shown in Fig. 5/b.

The previously mentioned facts concerning the flow curves are almost true for the results of hardness measurements. After the monotonic growing, from the fourth pass the cyclic increasing-decreasing trend is characteristic. The hardness values by the forged specimens are smaller than in the different stages of plain strain compression test, as it was demonstrated also concerning the flow curves.

The results of tensile test are summarized in Fig. 6. On the right graph the derived mechanical properties versus the characteristic summarized equivalent strains, calculated for one to ten times forged specimens, are plotted.
**Figure 6.** The tensile curves of plate samples manufactured from 1 to 10 times forged specimens (a) and the derived mechanical properties (b).

The results of tensile test are summarized in Fig. 6. On the right graph the derived mechanical properties versus the characteristic summarized equivalent strains, calculated for one to ten times forged specimens, are plotted. The values at zero strain represent the mechanical properties at the initial annealed state.

Analyzing the evolution of mechanical properties, the 0.2% offset yield strength ($R_{p,0.2}$) increases significantly up to the fourth forging pass, as it was shown concerning the flow curves. At the following states, after five, six, seven, eight and nine deformation pass the yield and the ultimate tensile strength ($R_m$) shows a decrease followed by slight increase. After the tenth pass, a significant growing can be observed again. Simultaneously the total elongation to fracture ($A$) decreases strongly after the first pass to the half of its initial value, however a moderated growing is indicated up to the sixth forging pass. If the strain continues to increase, the total elongation degrades, but it does not drop considerably below the corresponding value of first forging pass. The derived mechanical properties are summarized in Table 1.

| Initial state | Applied forging passes [-] |
|---------------|---------------------------|
|               | 1  | 2  | 3  | 4  | 5  | 6  | 7  | 8  | 9  | 10 |
| $R_{p,0.2}$ [MPa] | 86.0 | 142.5 | 141.2 | 147.0 | 165.3 | 150.3 | 149.5 | 155.8 | 156.3 | 150.7 | 183.9 |
| $R_m$ [MPa]     | 101.5 | 173.0 | 155.6 | 157.7 | 187.4 | 168.4 | 185.9 | 184.2 | 185.5 | 176.8 | 210.1 |
| $A$ [%]         | 45.4 | 17.1 | 25.1 | 18.9 | 21.4 | 23.5 | 23.9 | 19.3 | 21.2 | 22.5 | 16.1 |

TEM studies have presented that the 10-passes multiaxial forging process results in formation of mainly equiaxed microstructure with the grain and sub-grain size in the submicron range, as shown in Fig. 7.
Figure 7. TEM micrographs of ultrafine grained aluminum processed by 10-passes multiaxial forging.
In image (c) separated ultrafine grains can be clearly identified and the thickness contours in the grain boundaries show their relaxed and dislocation-free character. Moreover, the volume of these grains is also mainly dislocation-free. In the dark field image from the same area, three grains can be seen in close to each other Bragg-orientation but in the neighboring black area the orientation of grains is significantly different. Furthermore, in the diffraction pattern in image (a) the reflections are scattered on an arc of about 120 degrees that also proves the existence of high angle grain boundaries. Consequently, these bright grains are bounded by mainly high angle grain boundaries and they are probably formed during the deformation process through annihilation of dislocations indicated by the mentioned thickness contours. Close to these relaxed grains, dark areas can be observed in many groups, as one of them is demonstrated in the enlarged image (b). Possibly, these grains are formed by splitting due to the intensive deformation because their substructure contains several subgrains close to Bragg-position bounded by low angle grain boundaries. This fact is also confirmed by the selected area diffraction pattern in image (b), on which the reflections are in groups. The large black or white arrow shows the boundaries of cells composed of dislocations. It follows that these dark grains represent the intermediate stage of grain refinement, while the bright grains with the thickness contours have already relaxed at the end of the last forging pass. Considering the grain size, many grains were measured and the average is approximately 560 nm.

4. Conclusions

In this study the process of multiaxial forging simulation was modeled as well as the resulted microstructure and the evolution of different mechanical properties was investigated by experiments on MaxStrain System using Gleeble physical simulator. A new mechanical model was developed based on the principle of virtual velocities using the continuous measurement of the dimensions on the deformed volume as well as the derived area of contact surfaces. In this mechanical model the actual flow stress was calculated at the stationary point of the functional of plasticity. The evolution of calculated flow curves is significantly different from the flow curve of monotonic deformation obtained by plane strain compression test, except of by the first forging pass at which the calculation resulted in almost the same flow stresses. The odd and even forging steps can be separated, reflected in the cyclic alternately growing of flow stress and the different enveloping curves described by the same formula but showed significantly difference respect to the hardening exponents. The statistical analysis of hardness measurements is consistent with the evolution of calculated flow curves. In the beginning the hardness increases as the strain begins to accumulate in the deformed volume. Further increase in the number of deformation passes resulted in a decreasing-increasing change of hardness. Comparing the maximum flow stress obtained by the mechanical model and by the calculation based on the median of hardness values, the difference is less than 12% at each forging pass. The mechanical properties derived from the tensile tests indicate significant growing in strength and gradually eliminating decrease concerning the toughness. The TEM studies showed, the formed microstructure is ultrafine grained and most of grains have relaxed and dislocation-free character with equiaxial shape and quite uniform grain size distribution with a grain size near to 500 nm.

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