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MODELING WITH FCA-BASED MODEL OF MICROSTRUCTURE EVOLUTION OF MgCa08 ALLOY DURING DRAWING OF THIN WIRE IN HEATED DIE

MODELOWANIE ZA POMOCĄ FCA ROZWOJU MIKROSTRUKTURY STOPU MgCa08 PODCZAS CIĄGNIENIA CIENKIEGO DRUTU W PODGRZEWANYM CIAGADLE

The paper deals with a modeling of manufacturing process of thin wire of MgCa08 alloy used as biocompatible soluble threads for medical application. Some difficulties in material deformation subjected with its hexagonal structure can be solved with accurate establishment of the deformation conditions, especially temperature history of the whole process. In drawing process with heated die, wire is preheated in furnace and then deformed. The only narrow temperature range allows for multi-pass drawing without wire breaking. Diameter below 0.1 mm required for the final product makes very important the consideration of microstructure evolution because grain size is comparable with the wire dimensions. For this reason the problem is considered in the micro scale by using the frontal cellular automata (FCA)-based model. The goals of present work are the development and validation of FCA-base model of microstructure evolution of MgCa0.8 magnesium alloy. To reach this objective, plastometric and relaxation tests of MgCA08 alloy were done on physical simulator GLEEBLE 3800. Results of the experimental studies were used for parameters identification of the hardening-softening model of the material. Then, initial microstructure and its evolution during the drawing passes were simulated with FCA-based model. FCA consider dislocation density and flow stress, hardening and softening including recovery and recrystallization, grain refinement and grain rotation, as well as grain growth. It allows one to obtain structures close to real ones. Two variants of the drawing process with different temperature history were considered. The deformation scheme was the same. Simulation results with following short discussion confirm usefulness of FCA-based model for explanation and selection of rational technological condition of thin wire drawing of MgCa08 alloy.

W pracy rozpatrzono proces wytwarzania cienkich drutów z biozgodnego stopu MgCa0.8 z przeznaczeniem na resorbowalne nici chirurgiczne. W procesie ciągnienia drut nagrzewany jest w piecu a następnie odkształcany. Jednym z warunków, jaki musi być spełniony w technologicznym procesie jest zachodzenie rekrystalizacji w trakcie ciągnienia. Pozwala to na realizację wielo przepustowego procesu ciągnienia bez wyżarzania międzyoperacyjnego. Prognozowanie rekrystalizacji na etapie projektowania technologii wymaga stworzenia modelu rekrystalizacji. W przypadku ciągnienia drutów o średnicach mniejszych niż 0.1 mm konieczne jest zastosowania modelu w skali mikro. Celem pracy jest opracowanie modelu rekrystalizacji, opartego o frontalne automaty komórkowe (FCA) oraz przykładowa symulacja kilku przepustów ciągnienia. Do kalibracji modelu FCA wykorzystano badania plastometryczne oraz testy relaksacji stopu MgCa08 przy użyciu symulatora fizycznego GLEEBLE 3800. Wyniki tych badań pozwoliły wyznaczyć parametry modelu umocnienia-mięknięcia materiału. Następnie początkowa mikrostruktura i jej rozwój podczas procesu ciągnienia były analizowane za pomocą modelu opartego o FCA, który uwzględnia gęstość dyslokacji, naprężenie uplastyczniające, umocnienie i mięknięcie w tym zdrowienie i rekrystalizację, rozdrobnienie ziaren oraz ich rotację i rozrost, co pozwala na uzyskanie struktury bliskiej do rzeczywistej. Dwa warianty procesu ciągnienia o różnej historii zmiany temperatury rozpatrzono w pracy. Wyniki symulacji potwierdziły przydatność modelu opartego o FCA do uzasadnienia i wyboru racjonalnych warunków technologicznych ciągnienia cienkich drutów za stopu MgCa08. W pracy przedstawiono również praktyczną implementację procesu ciągnienia.

1. Introduction

Magnesium alloys are applied in medicine for sake of high compatibility and solubility in human organism [1,2]. Production of surgical threads used for integration of tissue may be one of the applications of those alloys types [2,3]. This application requires wires with diameter about 0.1 mm. Magnesium alloys are very difficult for deformation, which is connected with their hexagonal close packet structure. That is why a new manufacturing technology of wire drawing process in heated die assisted with numerical model was developed [4,5]. This technique is designee especially for thin

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magnesium wires with diameter less then 1.0 mm. According to this technology the drawing process performed in conditions where the recrystallization occurs in every draft. In this case it is possible to carry out multi-pass process without annealing.

Model of recrystallization of MgCa0.8 magnesium alloy in macro scale was developed and presented in [6]. This model allows for quick prediction and optimization of drawing. However, the results presented in [7,8] suggest that the impact of the thermo-mechanical processes, occurring in the micro scale is essential, especially for the thin wire. For this reason, in the present study, the recrystallization process is considered in the micro scale using the cellular automata (CA) model.

CA models are very suited for the study of microstructure evolution. CA are the main element of digital material representation, which are developed dynamically nowadays. The CA applications for simulations of different phenomena are very important in the materials science, and their role is increasing. CA are used for modelling of crystallization (solidification) [9–14], dynamic and static recrystallization [15-19], phase transformation [17, 20–23], cracks propagation, grain refinement [24-26], etc. Complex microstructural phenomena and technological processes are modelled on the base of CA. Interaction between solidification and deformation, recrystallization and phase transformation can be considered and modelled by CA [27-29].

A hierarchical system based on FCA for modelling of microstructural phenomena and technological processes was developed [27]. The basis of this system is FCA, while the top is a set of processes. Models of microstructural phenomena are between them and connect them. The database of materials completes the system. FCA and their computational advantages are described in detail elsewhere [19, 21, 23, 27]. Consideration of the deformation in FCA is described as well [21, 30].

Description of some modules of the system can be found in previous publication: initial microstructure [31], crystallization [14], recrystallization [19, 21], phase transformation [23] and grain refinement [27]. Results of modelling and simulation with this FCA-based system are presented elsewhere: solidification in continuous casting [14], hot flat and shape rolling [21, 30], the roll-bonding process and MAXStrain technology [27, 32], powder bed generation [33], etc.

The goals of present work are the development and validation of FCA-base model of microstructure evolution of MgCa0.8 magnesium alloy.

2. Experimental study of MgCa08 alloy

Properties of MgCA08 were studied with physical simulator GLEEBLE 3800. Relaxation tests were carried out. During the tests, specimen was heated from the room temperature to the temperature of deformation with the rate 20 K/s, then it was withstood for 5 s and deformed with rate 1 s^{-1} to preset strain. After the deformation, temperature is kept up the same as during the deformation, strain rate is dropped to the value near zero to keep specimen in the tools and to measure stress that decreasing with the time because of relaxation. Nine tests were fulfilled with all variants of three values of temperature – 250, 300 and 350ºC and three values of strain – 0.1, 0.2 and 0.3. Measured stress was used for both stress-dislocation and relaxation models. Results of identification of models parameters are described below

2.1. Dislocation density and flow stress model

FCA should use the model of dislocation density for proper simulation of microstructural phenomena that take place in material during the thermo-mechanical processes. Flow stress is closely connected with the dislocation density and allows to identify parameters of dislocation density model on the basis of plastometric tests. Flow stress can be treated as a bridge between the dislocations and their macroscopic effect during the deformation.

Review on dislocation density and flow stress models for magnesium alloys can be found in Trojanova and Lukac [7]. They considered models developed by Kocks [34], Estrin and Mecking [35], Malygin [36], Nes and Marthinsen [37] and other.

The flow stress model used in FCA is based on general internal variable ρ_{av} that can be treated as dislocation density. Theoretical basis of the model was developed and described in previous publications [38-41]. But this model was developed for metals with bcc lattice and should be corrected for magnesium alloys with hexagonal close packed (hcp) structures. The following equation describes the flow stress as a function of the general internal variable ρ_{av}:

$$\sigma = \sigma_0 + \alpha\mu b\sqrt{\rho} \tag{1}$$

General internal variable ρ depends on three simultaneous processes. Evolution of dislocation density interior the dislocation cells structure (subgrains and fine grains), evolution of cells structure into the subgrains and grains, i.e evolution of dislocation density in cells walls, and recrystallization. All processes are evolving in the background of recovery:

$$\rho = \rho_1 + \rho_2 - \rho_3 \tag{2}$$

Then, we consider following three equations.

$$\rho_1 = A_1 \exp\left(\frac{Q_1}{RT}\right)\left[1-\exp\left(-\frac{\varepsilon}{\varepsilon_1}\right)\right] \tag{3}$$

$$\rho_2 = A_2 \exp\left(\frac{Q_2}{RT}\right)\left[1-\exp\left(-\frac{\varepsilon}{\varepsilon_2}\right)\right] \tag{4}$$

$$\begin{cases} 0 & \forall\, \varepsilon \le \varepsilon_c \\ \rho_3 = A_3 \exp\left(\dfrac{Q_3}{RT}\right)\left[1-\exp\left(-\dfrac{\varepsilon-\varepsilon_c}{\varepsilon_3}\right)\right] & \forall\, \varepsilon > \varepsilon_c \end{cases} \tag{5}$$

The first and second equations describe the fast and slow dislocation density growth, connected with dislocation increasing into dislocations cells and in the walls of dislocation

cells. These equations describe changes of dislocation density during the deformation and they consider results of two processes – hardening (dislocations generation and pilling-up) and recovery (annihilation of dislocations). The third equation describe dynamic recrystallization that appear after strain reaches a critical value ρc. Parameters were identified on the base of relaxation tests, namely on the first part of these tests when specimens were deformed. The obtained parameters are following:
A_1 = 0,0711; A_2 = 258; A_3 = 422; Q_1 = 38630; Q_2 = 9920; Q_3 = 4400;
ε_1 = 0,0096; ε_2 = 0,243; ε_3 = 0,00224T-0,519; ε_c = 0,00743-3,8×10^{-6}T.

Results of parameters identification can be presented in comparison with experimental data. Good coincidence was obtained, it can be seen in Fig. 1. In this figure, the empty symbols represent experimental data, while filled symbols are model results. It should be mentioned, because of tests conditions, the usefulness of the model is limited with temperature 250 to 350ºC and strain rate 1 s^{-1}. Though, we allow ourselves to extrapolate results to 390ºC and flow stress predicted for this temperature is shown in the fig. 1 as well.

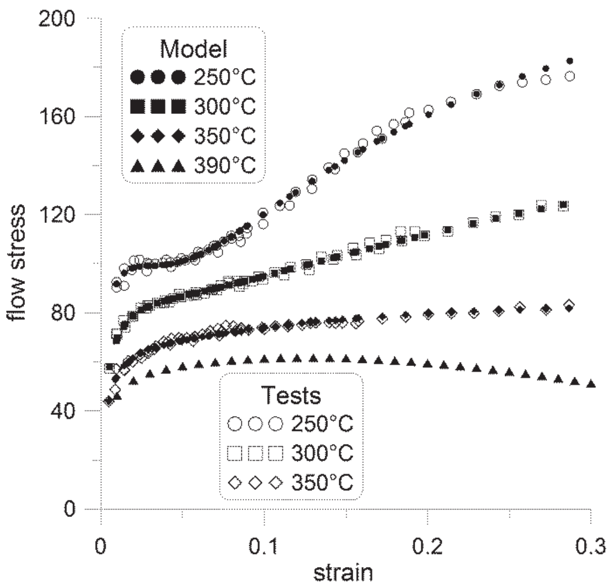


Fig. 1. Flow stress of MgCa08 alloy: empty symbols represent experimental data, filled symbols are model results

Some decrease of flow stress on the beginning section of the curve can be probably observed for low temperature (below 250ºC). The lowest temperature is the deepest and widest decrease is. It leads to localization of deformation, and breaking of the wire can appear during the drawing if temperature is low enough [4]. The other results are obtained at higher temperature (over 350ºC). Flow stress continuously decrease after it reach the maximal (critical) value. Simultaneously, the cross-section of the wire decreases, it leads to extreme increase of the tensile stress, strain rate in appropriate localization. Then, positive feedback switch on and wire is broken. An example of such a fracture mechanism is described in [5].

2.2. Relaxation model

Stacking-fault energy (SFE) of magnesium alloys is 125-135 mJ m^{-2}, between SFE of copper (70-78) and aluminum (160-200). It is well known the recrystallization appears easily in the higher temperature and higher stored deformation energy in materials with low SFE. In the materials with high SFE, the static recovery dominate over the recrystallization. The magnesium alloys occupy medium place. Studies of annealing process [5] for AZ31 show the recrystallization temperature depends on strain and varies 190ºC to 140ºC when strain change from 0.1 to 0.3. For the same strains, temperature, when grains begin to grow, is 230 to 160ºC. Thus, the considered drawing process is probably provided at the temperature, at which three softening processes take place simultaneous. They are recovery, recrystallization and grain growth.

Nes [42] reviewed static recovery. There are two models considered and compared. Nes stressed that dislocations of opposite signs may react due to glide and climb. Then he noted that any one of these reactions: glide, cross-slip or climb can be the rate controlling step in the growth of a dislocation network. According to Nes model [42], the dislocation density varies in time as

$$R = \sqrt{\frac{\rho}{\rho_0}} = \left[1 + \frac{t}{\tau}\right]^a \tag{6}$$

where a = l/2 for vacancy bulk diffusion, a = l/4 for vacancy core diffusion, with lateral drift a = l. When lateral drift is not considered can be used following equation:

$$\sqrt{\frac{\rho}{\rho_0}} = 1 - \frac{kT}{A}\ln\left(1 + \frac{t}{\tau}\right) \tag{7}$$

Combining different recovery mechanisms the fraction residual strain becomes:

$$R = f\left[1 + \frac{t}{\tau_1}\right]^{a_1} + (1-f)\left[1 + \frac{t}{\tau_2}\right]^{a_2} \tag{8}$$

In equations (6) –(8) t is current time, τ is relaxation time parameter, f – fraction.

Some parameters of the models (6)-(8) can be found in [42].

Martin et al [8] consider continuous and discontinuous recrystallization in magnesium alloys. Accumulation and rearrangement of dislocations leads to the formation of numerous low-angle boundaries (LABs) while strain increase. Subsequent rotation of the new substructures results in appearance of more and more high angle boundaries (HABs). This process is generally referred to as continuous dynamic recrystallization (cDRX) or extended recovery. Nucleation by the bulging of initial grain boundaries can appear in magnesium alloys as well. It leads to discontinuous dynamic recrystallization (dDRX) and metadynamic recrystallization. Static recrystallization is not usually considered because of high rate of recovery.

Basing on condition of modeled process, when high temperature lasts for very short time, the third mechanism of softening, i.e. grain growth, is neglected.

Then, equation (8) is used for model of recovery. Parameters identification of the model was fulfilled on the data received from the Gleeble 3800 during the relaxation test. Simplified linear model for parameters (8) is applied:
$a_1 = -0.5$, $a_2 = -1.0$, $f = -2.728 + 0.0059T - 0.524\varepsilon$,
$\tau_1 = 3.0217 - 0.001625T - 5.464\varepsilon$,
$\tau_2 = 0.1294 - 1.346\times10^{-5}T - 0.2717\varepsilon$.

Results of identification models parameters shown in Fig. 2 demonstrate a good coincidence.

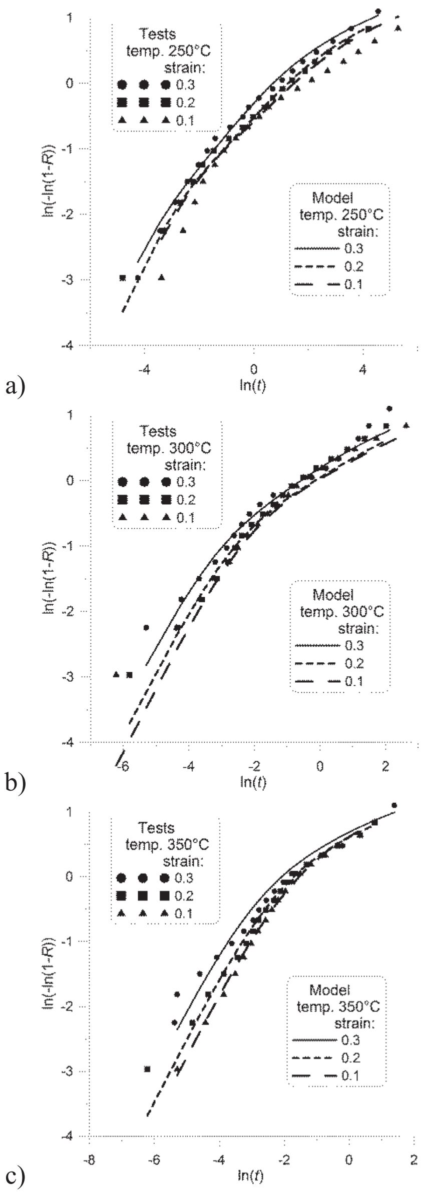


Fig. 2. Comparison of flow stress relaxation model with tests (strain rate 1 s-1): a - 250ºC, b - 300ºC, c - 350ºC

2.3. FCA simulation

The technological process of manufacture of thin magnesium wires contains 25 passages. Initial wire diameter is 1 mm and a final diameter equal to 0.1 mm. Detailed description of the technology is given in [5]. For the last pass, when the wire diameter becomes small, the analysis recrystallization in micro scale is particularly important. For this reason, the last two passes of the drawing process were simulated. Diameter of the wire before and after the first pass is 0.123 and 0.112 mm correspondently. Diameters of wire in second pass are 0.112 and 0.1 mm. The wire entered into the furnace, where is heated up to deformation temperature (300-320ºC).

Two variants of drawing process with velocity: 10 and 60 mm/s were simulated. After the deformation, wire is moving out from the tools and furnace and then is cooling down in the air to temperature 20ºC.

Total time, when the wire is into the furnace, depends on drawing velocity and equals to 14 and 3.33 s (length of furnace was 140 mm). Simulation of wire drawing was performed with help of Drawing2d FEM program [7]. Simulation results of drawing process showed that the gradient of temperature in a cross section of wire is very small (fig. 3,a). For this reason the temperature change of metal during the drawing process can be characterized by the average value of the temperature over the cross section. The simulation results for all 25 passes are shown in fig. 3,b. Temperature calculated for the last two passes were used in the FCA simulation.

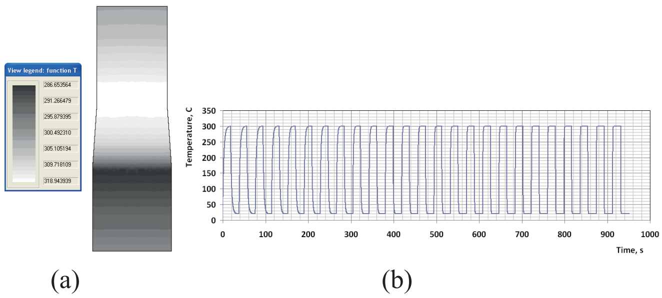


Fig. 3. Distribution of temperature in deformation zone during drawing in last pass (a) and distribution of average temperature in cross section of wire during 25 passes (b) for drawing velocity 10 mm/s

Analyzing the simulations and measurements of the temperature changes, it was noted that temperature distribution on crass-section can be neglected. Heating and cooling during the last two passes can be approximated in the same manner. Then, in FCA, average temperature during heating and cooling can be approximated in time with the following ordinary differential equation:

$$\frac{dT}{dt} = \frac{1}{\theta}\left(T_\infty - T\right) \quad \forall \, T\big|_{t=0} = T_0 \tag{9}$$

where: T – current temperature, T_0 – initial temperature, T_∞ - final temperature (temperature of furnace or air), θ – time constant. Analytical solution of (9) is of following form for heating and cooling (t – time from the wire entrance into the furnace or its exit):

$$T = \left(T_\infty - T_0\right)\left[1 - \exp\left(-\frac{t}{\theta}\right)\right] + T_0 \tag{10}$$

For heating the following values can be set for the variables and parameters:
$\theta = 0.3$ s, $T_0 = 20$ºC, $T_\infty = 300$ºC
For cooling, they are the next:
$\theta = 0.53$ s, $T_0 = 300$ºC, $T_\infty = 20$ºC

Equation (10) with above mentioned parameter gives maximal error about 4ºC compared with the data shown in the figure 3,b, that is acceptable for further simulations.

Equation (8) was applied for condition of drawing described by (9) and (10). Fraction of softening is presented in figure 4. During the cooling dislocation density decreases 4 times and remains in this level till the next entrance. Time of heating before next drawing pass depends on drawing velocity and varies 2.35 to 14 s. Then, residual relative hardening equals 0.08 to 0.02 for such conditions and it can be neglected.

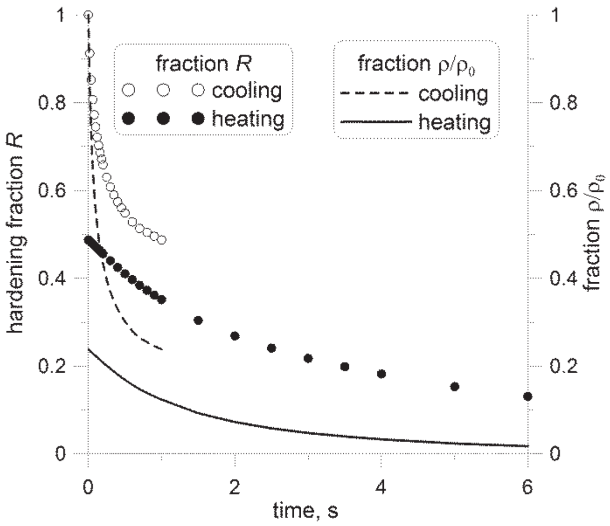


Fig. 4. Simulation of relaxation in pause between passes

2.4. FCA simulation

Initial microstructure for simulations is created basing on following assumption.

The first entrance of the wire into the furnace does not effect on microstructure because it is lack of appropriate information needed for simulation of recrystallization or recovery. Then wire enters into the die, where it is deformed and reach new diameter. FCA simulate drawing. Dimensions of representative volume are changed, flow stress and dislocation density are calculated. Inside the grains, a creation of new dislocation cells is modeled. As it was demonstrated in [31, 32], dislocation cells appear at the beginning stage of severe plastic deformation. Then the dislocation cells evolves in subgrains and new grains. Difference between dislocation cells, subgrains and new grains can be expressed by boundaries disorientation angle. LABs characterize dislocation cells, while HABs depict grains. Because the last passes of drawing process are modeled, the final microstructure is mainly formed far before, i.e. the most of dislocation cells, which appeared in the first passes, evolved into subgrains and grains. Grains were refined and almost reach minimal value appropriate for thermomechanical conditions of multi-pass drawing process. It is assumed that grains during the deformation in the pass are elongated and decreased radial cross-section. At the same time, crystal lattice strives to maintain its optimal orientation achieved in the previous passes. Conditions of cDRX are remained for grains extended circumferentially only. Nucleation for dDRX can appear all the time, and it is taken into account, though by the last passes its intensity decrease almost to zero. The recovery is assumed to be main relaxation mechanism in this stage of drawing. Nevertheless, cDRX and dDRX are considered in simulations. It should be added, time of grain growth during dDRX is very limited by the fast recovery.

Microstructures before passes, between two passes and after the drawing are presented in the figures 5. Initial microstructure for simulation was created according to conditions and principles described in [31].

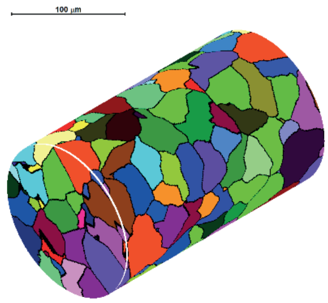
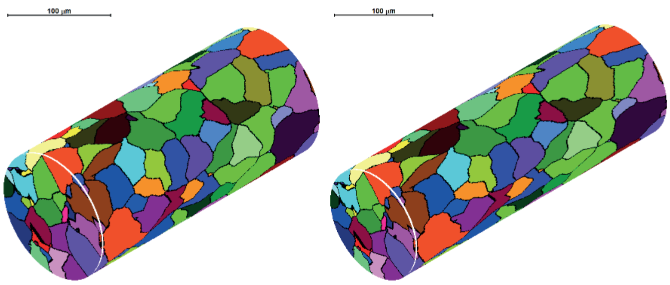


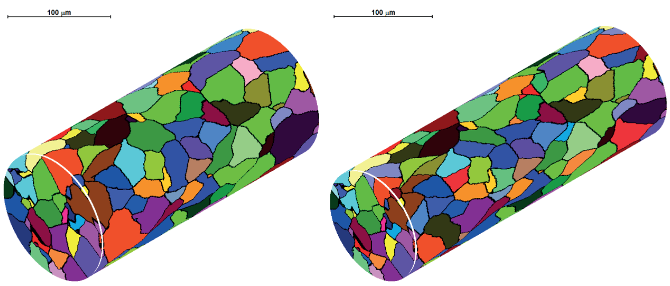
Fig. 5. Initial microstructures before passes

Two variants microstructure after the last two passes a presented in figures 6-7 for comparison. The first microstructures (fig. 6) demonstrate only grain deformation without consideration of microstructure evolution. .The second microstructures (fig. 7) were obtained during the modeling of the drawing process with simulation of cDRX. Effect of cDRX can be observed for several grains, which were split on parts, while the most others saved their shapes and sizes. Some refinement is the result of cDRX.



a) b)

Fig. 6. Microstructures before (a) and after (b) the last pass without microstructure evolution



a) b)

Fig. 7. Microstructures before (a) and after (b) the last pass with microstructure evolution

3. Experimental validation

The drawing process was validated with experimental equipment [43] shown in Fig. 8. Wire diameter was reduced in two drawing passes from 0.123 mm to 0.112 and 0.100 mm; furnace temperature was 320°C; drawing velocity was 10 and 60 mm/s. The experimental process was stable without fracture during the drawing with velocity 10 mm/s. Analysis of the material after last passes showed that the wire material is flexible (Fig 9a) and contains only recrystallized grains (Fig. 9b). In the second variant (60 mm/s) after two passes material was not flexible enough that is associated with incomplete recrystallization.

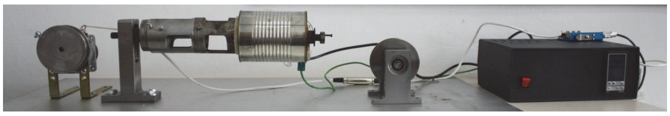
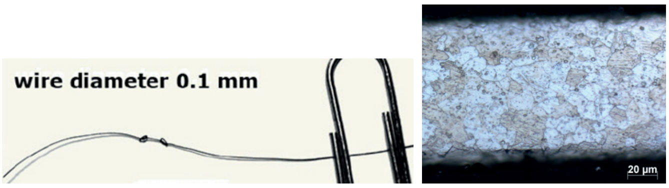


Fig. 8. The experimental device used to perform the drawing process in the hot die



a) b)

Fig. 9. The wire d = 0.1 mm after drawing according to drawing velocity 10 mm/s, the general view (a) and the microstructure (b) [5]

4. Conclusions

In the paper, results of plastometric tests of MgCa08 fulfilled on physical simulator GLEEBLE 3800 were presented and analyzed. The results allow to explain why multi-pass drawing without wire breaking can be provided in the narrow temperature range only. The higher temperature is limited with strain when appear negative slope on the strain-stress curve. For the temperature of 350ºC, this strain is about 0.15. At the temperature below 250ºC, the negative slope can be observed at the very beginning, and it can be cause of stress and strain localization that leads to wire breaking.

Application of appropriate model for flow stress allows us to obtain good agreement between experimental data and value obtained with the model.

Results of relaxation tests help us to choice model of relaxation and softening. Parameters of relaxation model were identified, and model verification demonstrated good compliance with experimental study. Analysis of softening kinetics showed that natural cooling of the wire in air after the drawing pass enough for 50% material softening (dislocation density decreases four times), and during the heating before next pass softening process is mainly completed (12-20% residual hardening or 2-8% of dislocation density during the deformation).

The fast recovery (or cDRX) of MgCA08 alloy in conditions of modeled process makes effects of dDRX insignificant, thought it remains in the model.

Then, initial microstructure and its evolution during the drawing passes were simulated with frontal cellular automata based model. Two variants of microstructure were presented to show effect of drawing in the last two passes on final microstructure and its evolution.

Validation of presented model was done using experimental installation designed for drawing process in heated die. Results of drawing process are consistent with the numerical model which proves that the model parameters were correctly designed.

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