Comparison between different methods to determine material constants of the ZK60 Mg alloy from hot bulge tests data

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Abstract. ZK60 is an Aluminium-free Magnesium alloy able to achieve high ductility in superplastic conditions. Those properties make the alloy suitable to be investigated for temporary prostheses production by means of the Superplastic Forming (SPF) process. To properly design the manufacturing process by numerical simulations, an accurate material constitutive model is needed. In the present work, bulge tests at 400°C were conducted on a ZK60 Magnesium sheet using different loading conditions, namely two different levels of constant pressure (CP) and jump pressure (JP) between the two pressure levels. The dome height evolution was acquired during each test and used to calibrate the material constants of the Backofen constitutive equation ($\sigma = C \cdot \dot{\varepsilon}^m$) using three different methodologies: (i) an analytical approach (ii) an inverse methodology based on a single CP test and (iii) an inverse methodology based on the JP test. The obtained constants were validated by subsequent numerical simulations. Comparing the numerical/experimental dome height curves, it was found that the sets of constants determined using the inverse methodology based on the JP test are able to describe the material superplastic behaviour over a wider span of loading conditions.

Introduction

Magnesium (Mg) based implants are gaining interest for temporary prostheses applications, since Mg mechanical properties are more similar to that of natural bones than the other metallic materials commonly used for implants, such as titanium alloys and stainless steels [1]. The ZK60 magnesium alloy can represent a viable alternative to the well known AZ31 for biomedical applications, since it is much safer for the human body due to the almost absence of Aluminium (Al < 0.05%), which is well-known for its toxicology related to neurodegenerative diseases (e.g., the Alzheimer disease) [2,3]. Furthermore, ZK60 showed good superplastic ductility in warm and hot conditions [4,5]. This ability to achieve high deformation make the alloy suitable to be processed by means of the Superplastic Forming (SPF) process [6], that uses gas pressure to form the sheet metal blank according to the die cavity shape. The resulting flexibility makes this technology suitable for the manufacturing of highly customised products, such as prostheses [7,8].

The adoption of the numerical approach allows to determine the gas pressure profile able to optimally exploit the material superplastic behaviour [8]. This requires a reliable FE model, based on an accurate constitutive material behavior. Tensile tests are commonly used to characterize materials superplastic behavior [9]. However, bulge tests can effectively reproduce strain conditions close to the ones occurring during the SPF process [10]. There are several types of bulge

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tests available in literature, e.g., the Constant Pressure (CP) test [11], in which a constant gas pressure is applied and the Jump Pressure (JP) test [12], in which a stepped pressure profile changing between two values is used. The experimental data in terms of dome-height evolutions according to time and the thickness profiles at certain instants are then used for determining the material constants according to different constitutive equations, by using analytical methods or by inverse analysis [13].

In the present work the ZK60 magnesium alloy (Zr 0.85%, Zn 5.56%) was investigated using both CP and JP tests. The dome height evolutions according to time were acquired and used for calibrating the material constitutive equation. In particular, the behaviour of the alloy in superplastic conditions was modelled using the Backofen power law (Eq. 1):

$$\sigma = C \cdot \dot{\varepsilon}^m \tag{1}$$

where σ is the equivalent stress, $\dot{\varepsilon}$ is the equivalent strain rate, C is the strength coefficient and m is the strain rate sensitivity index. Three different methodologies were used to obtain the two constants of the model (C and m): (i) by means of the analytical approach described in [11]; (ii) by means of an inverse methodology based on single CP tests and (iii) by means of an inverse methodology based on a single JP test [13]. The effectiveness of the approaches was finally evaluated by means of further numerical simulations using different loading conditions. In this way, the best set of material constants and the most effective methodology to determine them could be evaluated.

Material and methodology

Material. Bulge tests were performed on circular Mg alloy ZK60 specimens with a 75 mm diameter and 1 mm thickness. The chemical composition of the investigated alloy is reported in Table 1.

Table 1 Chemical composition limits [Weight %] of the investigated Mg alloy ZK60

Element	Al	Zr	Cu	Zn	Mn	Si	Ni	Fe	Mg
Content	0.0487	0.85	0.041	5.558	0.01	0.046	0.0045	0.00399	Bal.

Experimental bulge tests. Figure 1 shows the experimental setup used for conducting hot bulge tests. The circular Mg specimen was clamped between the blankholder and the die. Then, Argon gas was used to deform the ZK60 Mg alloy blank at 400°C. Argon pressure was controlled by a proportional valve (0-10V). To reach the target temperature, a solid-state induction heating system managed with a Proportional–Integral–Derivative (PID) controller was used; temperature was constantly monitored by means of K-type wire thermocouples welded close to the test area. A BlankHolder Force (BHF) value of 12 kN was set to avoid gas leakages. A laser sensor was used to acquire the dome height evolution according to time.

Bulge tests were conducted under different loading conditions: (i) a constant pressure (CP) test, in which a constant gas pressure equal to 0.25 MPa was applied (indicated in the following as CP0.25), (ii) a CP test applying a constant gas pressure equal to 0.5 MPa (indicated in the following as CP0.5) and (iii) a Jump Pressure (JP) test in which a stepped pressure profile changing between the two values (0.5 MPa and 0.25 MPa) was used. Tests were carried out up to failure.

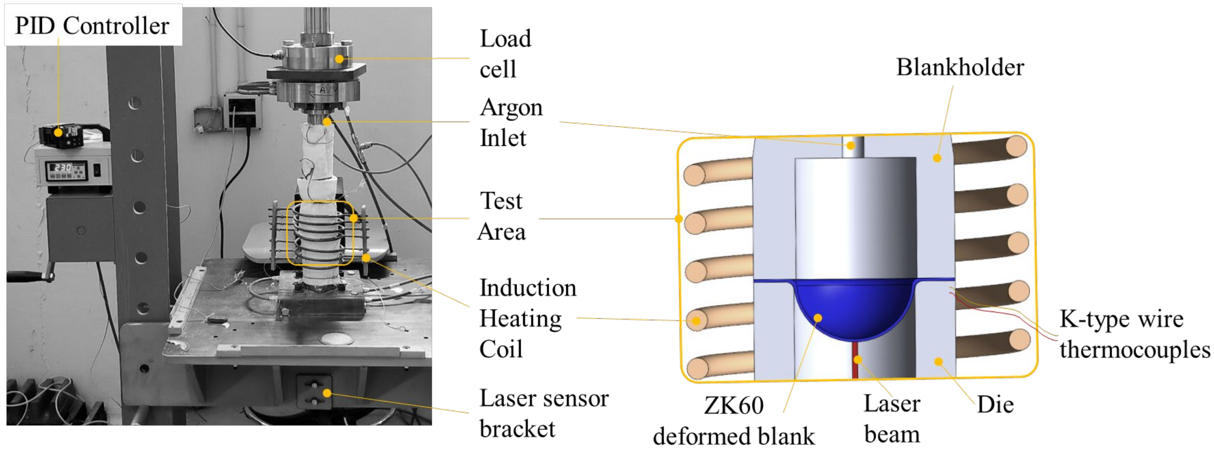


Figure 1: Bulge tests experimental setup

Analytical approach. The procedure proposed by Enikeev and Kruglov [11] was adopted for determining the material constants with an analytical approach. Data from dome-height evolutions of the experimental CP tests were used to determine the strain rate sensitivity index m as follows:

$$m = \frac{ln\frac{p_1}{p_2}}{ln\frac{t_2}{t_1}} \tag{2}$$

Where t_1 and t_2 are the forming times corresponding to the p_1 and p_2 pressure values (0.25 and 0.5 MPa, respectively). Forming times refer to a dome height target value (the same for both tests), falling into the constant slope portion of the dome-height curve. The strength coefficient was calculated as follows:

$$C = \left(\frac{p_f R_0}{2S_0}\right) \cdot [t_f/2I_m(\alpha)]^m \tag{3}$$

Where t_f is the forming time (referring to the target dome height value) at a constant pressure p_f, R_0 and S_0 are the die radius (22.5 mm) and initial thickness (1.0 mm) of the undeformed blank, respectively. The term $I_m(\alpha)$ is an integral that can be calculated as follows:

$$I_m(\alpha) = \int_0^\alpha [(sin^3x/x^2)]^{1/m} \cdot (1/x - cot\, x)dx \tag{4}$$

Where α is half the angle subtended by a dome surface at its center of curvature.

FE model for bulge tests. The commercial software Abaqus/Standard was used to numerically reproduce the testing conditions; to reduce the computational cost, an axisymmetric FE model was used; it is shown in Figure 2. The die geometry was modelled by means of an analytical rigid surface, whereas the ZK60 blank was meshed with 280 SAX1 elements having a size equal to 0.1 mm. The blankholder was not considered and its clamping action was simulated by blocking the translational degree of freedom along the radial direction. Friction between the blank and the die was modelled according to the Penalty formulation for contact and Coulomb friction and its coefficient was set to 0.1.

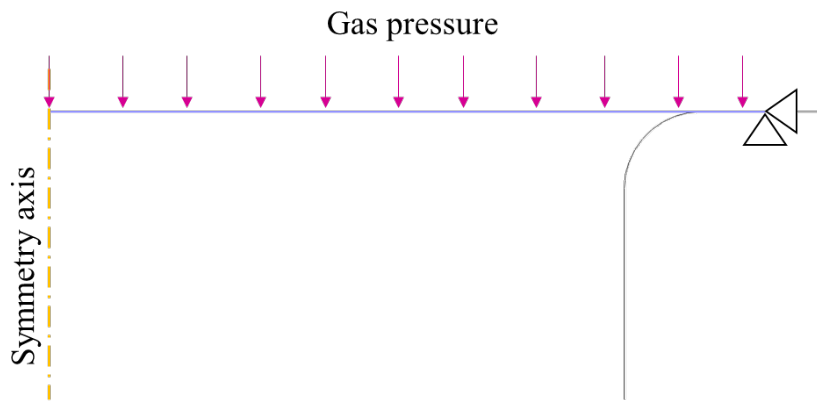


Figure 2: FE model used for the numerical simulations of bulge tests

The material behaviour was modelled using the Bailey-Norton power law ($\dot{\varepsilon} = A \cdot \sigma^n$), in which the constants (A, n) are related to the one of the Backofen power law ($\sigma = C \cdot \dot{\varepsilon}^m$) by the following equations:

$$n = {}^{1}/_{m} \tag{5}$$

$$A = \left({}^{1}/_{C}\right)^{1/m} \tag{6}$$

Pressure was applied to the upper surface of the blank. The dome-height evolution of the central point belonging to the bottom surface of the blank was recorded as output. The described numerical model was used both in the inverse analysis procedure and for validation purposes.

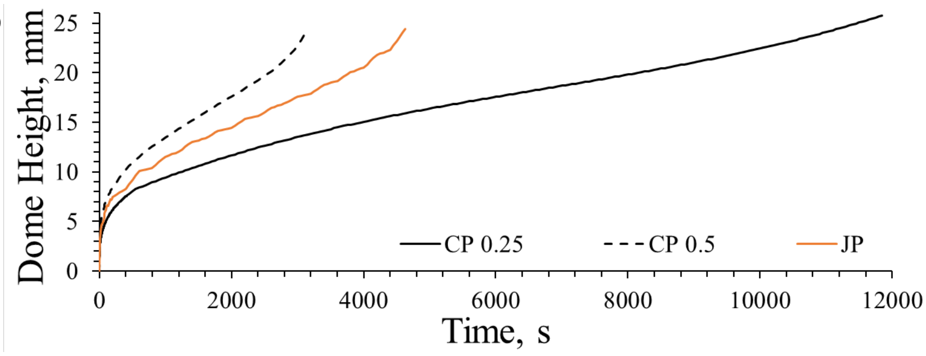
Inverse analysis approach. In the present work, material constants (C, m) in the Backofen constitutive equation (Eq. 1) were the unknown inputs to be determined by inverse analysis (IA). The numerical model described in the previous section was used to recreate the experimental working conditions; the experimental dome height curves were fitted by properly changing the value of the material constants (C, m) in the numerical simulations, minimizing the error between the numerical and the experimental data. The inverse analysis was driven by the MOGA-II genetic algorithm [14], that starts from an initial population created using the Sobol algorithm [15]. Additional details of the inverse analysis approach can be found in [13]. Each design represents a single numerical simulation characterized by specific values of the two constants (C, m); once each run was completed, the numerical evolution of the dome height was extracted by means of a python script file and written into an ASCII file. Subsequently, the numerical curve was compared with the experimental one and the sum of square residuals (SSR) calculated according to Eq. 7:

$$SSR = \sum_{i=0}^{N} \left(h_{i,num} - h_{i,exp}\right)^2 \tag{7}$$

Where N is the total number of measured time instants, $h_{i,num}$ and $h_{i,exp}$ are the numerical and the experimental value of the dome height at the i-th instant of time, respectively. Results were analyzed in terms of the history charts, able to describe the evolution of the input variables throughout the created generations.

Results and discussion

Experimental results. Figure 3 shows the experimental dome height evolutions of the ZK60 under the different pressure values, as well as the forming time and final height values.

(a)


(b)

Test ID	Gas pressure [MPa]	Forming time [s]	Final Height [mm]
CP 0.25	0.25	11843	25.7
CP 0.5	0.5	3146	24.3
JP	0.5 – 0.25	4631	24.4

Figure 3: (a) Experimental dome-height evolution of ZK60 during bulge tests at 400°C under different loading conditions (b) Loading conditions, forming time and final height of each test ID

Material constants from the investigated approaches. Three different methodologies were used to obtain the two constants (C and m) of the Backofen material model, namely by means of (i) the analytical approach, (ii) inverse methodology based on the CP0.25 and CP0.5 tests and (iii) inverse methodology based on the JP test. Values of C and m resulting from the analytical approach were $2224\ MPa * s^m$ and 0.63, respectively. For their determination a target dome height equal to 17 mm was considered, falling into the constant slope portion of both the CP 0.25 and the CP 0.5 dome-height curves as evidenced by Figure 3a.

Based on the results coming from the analytical approach, in the inverse analysis the strength coefficient C could initially vary between 100 and $4000\ MPa * s^m$, while the strain rate sensitivity index m between 0.3 and 0.7. The optimization loop was carried out considering an initial population of 50 individuals and 40 successive generations. Figure 4 shows the history charts resulting from the IA on the CP 0.25 test. Both C and m tended toward a stable and constant condition characterized by low SSR values after the 24th generation (i.e., after the ID 1200).

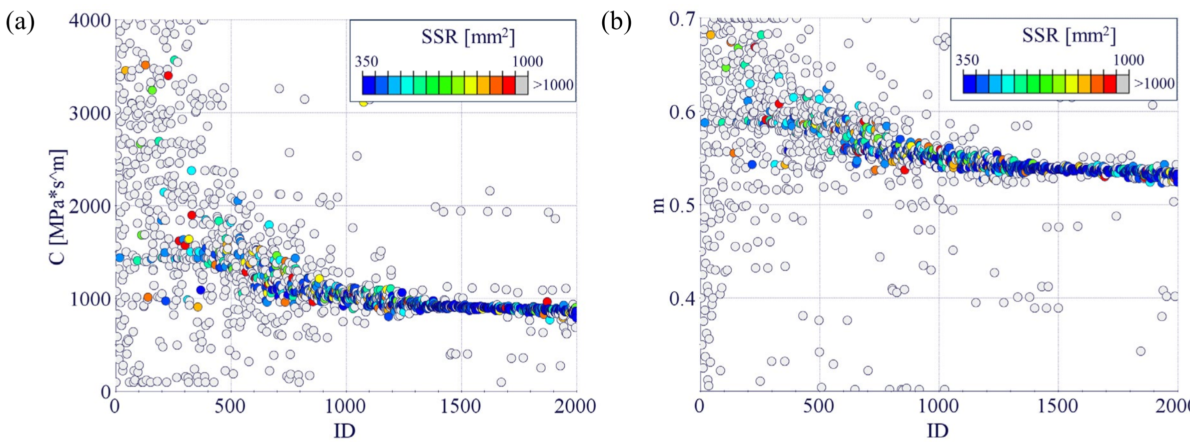


Figure 4: History charts for the IA on the CP0.25 test: a) strength coefficient C and b) strain rate sensitivity index m.

Figure 5 shows the history charts resulting from the IA on the CP 0.5 test. Unlike the previous case, C and m value tended toward a stable value almost at the end of the optimization loop, after the 32nd generation (i.e., after the ID 1600).

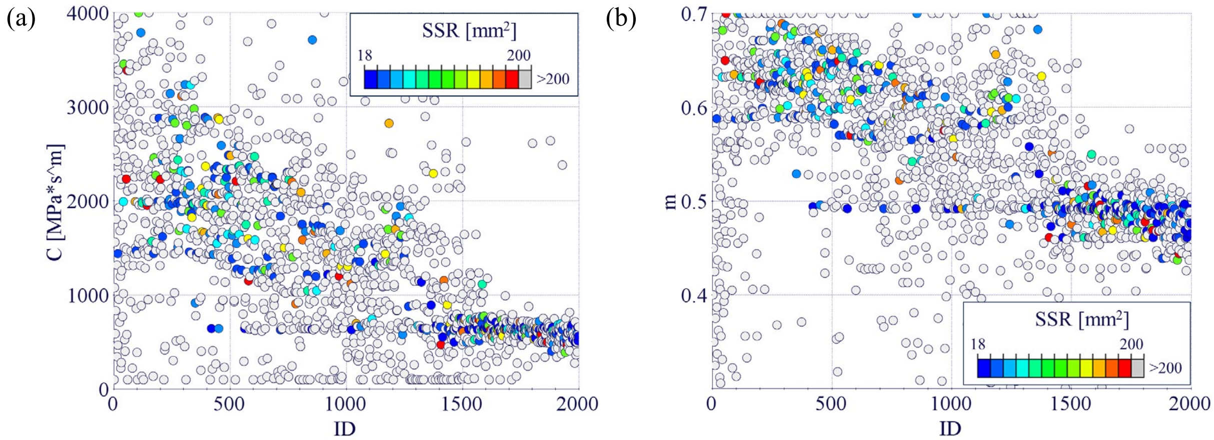


Figure 5 History charts for the IA on the CP0.5 test: a) strength coefficient C and b) strain rate sensitivity index m.

Figure 6 shows the history charts resulting from the IA on the JP test. After the 16th generation both the values of C and m tended already to a stable and constant condition, with minimum SSR values.

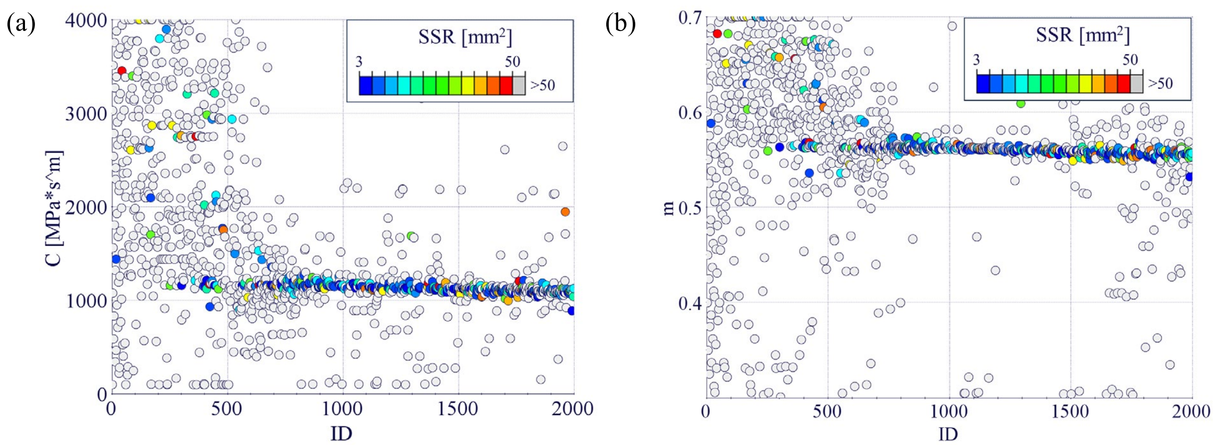


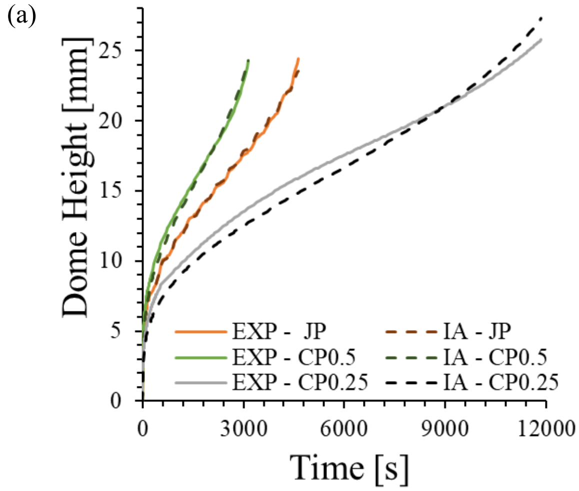
Figure 6 History charts for the IA on the JP test: a) strength coefficient C and b) strain rate sensitivity index m.

Figure 7a shows the comparison between the dome-height evolution of best design resulting from the IA and the experimental one (i.e., the target curve) for all the tested conditions. The best fitting of the target curve was obtained for the JP test, for which the input variables stably converged quicker. To compare results coming from different loading conditions (i.e., different forming times and, in turn, different numbers of measured time instants), the overall fitting was quantified by means of the Root Mean Square Error (RMSE), calculated as follows:

$$RMSE = \sqrt{\frac{\sum_{i=1}^{N}(h_{i,num}-h_{i,exp})^2}{N}} = \sqrt{\frac{SSR}{N}} \quad (8)$$

Where h_{exp} and h_{num} are the experimental and numerical dome height values and N is the total number of measured time instants. They are reported in Figure 7b. According to the results listed in Figure 7b, it appears that a lower applied pressure determined an increase of the calculated m value.

(a)



(b)

Test ID	C [MPa*s^m]	m	SSR [mm^2]	RMSE [mm]
CP 0.25	794	0.524	354	0.6
CP 0.5	496	0.461	18.5	0.2
JP	888	0.532	3.7	0.0

Figure 7: (a) Comparison between the dome-height evolution of best design resulting from the IA and the experimental one for all the tested conditions; (b) Material constants from the optimization runs, sum of square residuals (SSR) and Root Mean Square Error (RMSE).

Validation of the material constants. The previously determined constants able to describe the superplastic behavior of the ZK60 alloy were validated by means of further numerical simulations. In particular, the loading condition of the test CP0.25 was simulated implementing the material constants obtained (i) through the analytical (AN) approach (ii) by means of the inverse analysis carried out using data from the CP0.5 test and (iii) the set obtained through the inverse analysis on the JP test.

Figure 8 shows the numerical results in terms of dome height evolutions compared with the correspondent experimental ones (Figure 8a) as well as the RMSE between numerical and experimental dome-height curves (Figure 8b).

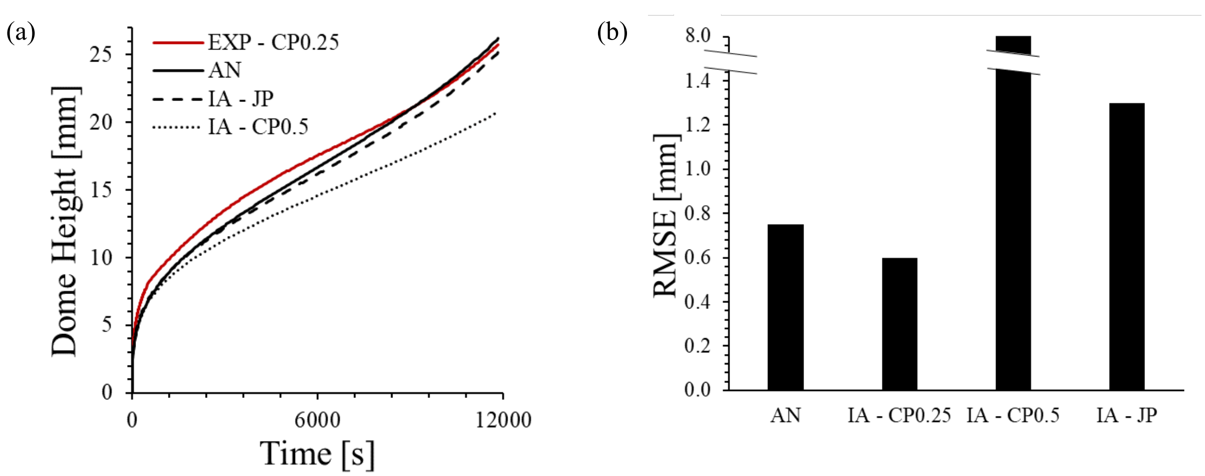


Figure 8: Validation runs on CP0.25 (a) Numerical/experimental comparison of dome height evolution (b) Root Mean Square Error (RMSE) between numerical and experimental dome-height curves

It can be noticed that implementing the material constants coming from the inverse analysis using data from the CP0.5 test (dotted curve in Figure 8a), the fitting was poor; in fact, as reported in Figure 8b, the RMSE value is close to 8 mm. This result suggests that when the constants are implemented to simulate a loading condition far from the one at which they had been determined using the inverse analysis, the fitting with the experimental dome height curve is not satisfactory. On the contrary, when implementing the material constants coming from the JP test (dashed curve), the fitting to the experimental data was better, with an RMSE value equal to 1.3. However, in this case a much better fitting was achieved when implementing the material constants determined through the analytical approach (solid black curve), that are characterized by a RMSE value equal to 0.7, slightly higher than the minimum value of RMSE (0.6), obtained when implementing the material constants coming from the inverse analysis using data from the CP0.25 test.

The loading condition of the CP0.5 test was similarly investigated implementing the material constants coming from (i) the analytical (AN) approach (ii) the inverse analysis carried out using data from the CP0.25 test and (iii) the set obtained through the inverse analysis on the JP test. Unlike the previous case, for the CP0.5 validation runs the constants calculated through the analytical approach provided the worst fitting (RMSE=1.3). On the contrary, the JP test also provided in this case a good fitting. In fact, the resulting RMSE (0.3) is slightly higher than the minimum value of RMSE (0.2), obtained when implementing the material constants coming from the inverse analysis using data from the CP0.5 test.

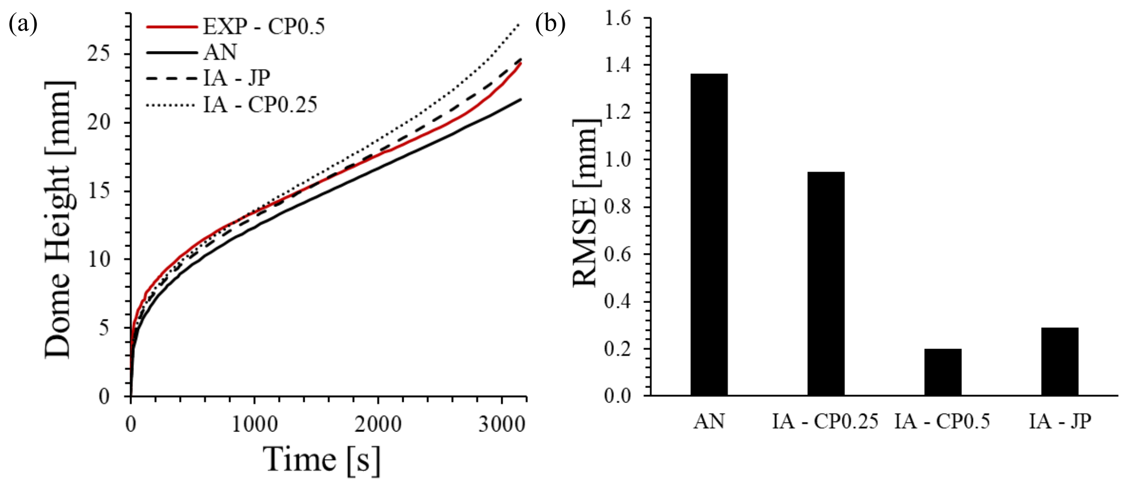


Figure 9: Validation runs on CP0.5 (a) Numerical/experimental comparison of dome height evolution (b) Root Mean Square Error (RMSE) between numerical and experimental dome-height curves

Considering both the validation runs cases (applying a bulging pressure of 0.25 and 0.5 MPa), it can be stated that the set of constants obtained through the inverse analysis using data from the JP test (IA-JP) showed a good fitting for both the pressure values, meaning that the constants were able to effectively describe the material behavior over a wider span of loading conditions. On the contrary, the set of constants obtained through the analytical approach (AN) provided a good fitting only at lower pressure values. Eventually, constants obtained by means of the inverse analysis using data from the CP tests (IA–CP0.25 and IA-CP0.5) can be representative only if used to reproduce loading conditions close to the ones at which they had been determined using the inverse analysis, otherwise they won't provide good results.

Conclusions

The present work investigates different methodologies for determining the material constants of the Backofen constitutive model able to describe the superplastic behaviour of the ZK60 alloy, starting from hot bulge tests data. Different sets of constants were obtained both through an analytical approach and by means of an inverse analysis approach. In this case, different sets of constants were obtained minimizing the difference between the numerical and the experimental dome height curves coming from both the constant pressure tests and the jump pressure test. The effectiveness of the approaches was evaluated by means of further simulations. Results showed that the set of constants determined by means of the inverse analysis approach using data from the jump pressure test ($C = 888\ MPa * s^m$ and $m = 0.532$) satisfactorily described in a wider pressure ranges the superplastic behavior of the ZK60 alloy in terms of dome height evolution. On the contrary, material constants determined using data from the constant pressure tests provided excellent results for loading conditions close to the one from which they had been determined, but they lost their capability to describe the superplastic behavior for different loading conditions. The analytical approach provided a set of constants able to well describe only one loading condition. In addition, unlike the inverse analysis approaches, it requires two different tests with different loading conditions for determining only one set of material constants. Thus, it can be stated that the most effective methodology to adopt for determining reliable ZK60 material constants of the Backofen constitutive model is the one based on the inverse analysis approach using data from the jump pressure test.

Future works will be aimed at expanding the investigation to different temperatures and loading conditions, as well as evaluating the reliability of the determined material constants considering the thickness distribution of the bulged specimens.

References

[1] B. Venkateswarlu, B.R. Sunil, R.S. Kumar, Magnesium based alloys and composites: Revolutionized biodegradable temporary implants and strategies to enhance their performance, Materialia 27 (2023) 101680. https://doi.org/10.1016/j.mtla.2023.101680

[2] J.R. Walton, Aluminum disruption of calcium homeostasis and signal transduction resembles change that occurs in aging and alzheimer's disease, Journal of Alzheimer's Disease 29 (2012) 255–273. https://doi.org/10.3233/JAD-2011-111712

[3] R.B. Heimann, Magnesium alloys for biomedical application: Advanced corrosion control through surface coating, Surf Coat Technol 405 (2021) 126521. https://doi.org/10.1016/j.surfcoat.2020.126521

[4] R. Lapovok, R. Cottam, P.F. Thomson, Y. Estrin, Extraordinary superplastic ductility of magnesium alloy ZK60, J Mater Res 20 (2005) 1375–1378. https://doi.org/10.1557/JMR.2005.0180

[5] W.J. Kim, M.J. Kim, J.Y. Wang, Superplastic behavior of a fine-grained ZK60 magnesium alloy processed by high-ratio differential speed rolling, Mat. Sci. Eng: A527 (2009) 322–327. https://doi.org/10.1016/j.msea.2009.08.064

[6] Z. Savaedi, R. Motallebi, H. Mirzadeh, R. Mehdinavaz Aghdam, R. Mahmudi, Superplasticity of fine-grained magnesium alloys for biomedical applications: A comprehensive review, Curr Opin Solid State Mater Sci 27 (2023). https://doi.org/10.1016/j.cossms.2023.101058

[7] P. Guglielmi, A. Cusanno, I. Bagudanch, G. Centeno, I. Ferrer, M.L. Garcia-Romeu, G. Palumbo, Experimental and numerical analysis of innovative processes for producing a resorbable cheekbone prosthesis, J Manuf Process 70 (2021) 1–14. https://doi.org/10.1016/j.jmapro.2021.07.060

[8] D. Sorgente, G. Palumbo, A. Piccininni, P. Guglielmi, S.A. Aksenov, Investigation on the thickness distribution of highly customized titanium biomedical implants manufactured by superplastic forming, CIRP J Manuf Sci Technol 20 (2018) 29–35. https://doi.org/10.1016/j.cirpj.2017.09.004

[9] F. Nazeer, J. Long, Z. Yang, C. Li, Superplastic deformation behavior of Mg alloys: A-review, Journal of Magnesium and Alloys 10 (2022) 97–109. https://doi.org/10.1016/j.jma.2021.07.012

[10] S. Aksenov, D. Sorgente, Determination of biaxial stress–strain curves for superplastic materials by means of bulge forming tests at constant stress, CIRP J Manuf Sci Technol 31 (2020) 618–627. https://doi.org/10.1016/j.cirpj.2020.09.002

[11] F.U. Enikeev, A.A. Kruglov, An analysis oft he superplastic forming of a thin circular diaphram, Int. J. Mech. Sci 37 (1995) 473–483. https://doi.org/10.1016/0020-7403(94)00081-T

[12] S. Aksenov, D. Sorgente, Characterization of Stress-Strain Behavior of Superplastic Titanium Alloy by Free Bulging Tests with Pressure Jumps, Defect & Diffusion Forum 385 (2018) 443–448. https://doi.org/10.4028/www.scientific.net/DDF.385.443

[13] A. Piccininni, D. Sorgente, G. Palumbo, Genetic algorithm based inverse analysis for the superplastic characterization of a Ti-6Al-4V biomedical grade, Finite Elements in Analysis and Design 148 (2018) 27–37. https://doi.org/10.1016/j.finel.2018.06.002

[14] S. Poles, E. Rigoni, T. Robič, MOGA-II performance on noisy optimization problems, Proc. Int. Conf. Bioinspired Optim. Methods Their Appl (2004) 51–62.

[15] P. Bratley, B.L. Fox, Implementing Sobol's Quasirandom Sequence Generator, ACM Transactions on Mathematical Software 14 (1988) 88–100. https://doi.org/10.1145/42288.214372