Journal of Metals, Materials and Minerals, Vol.19 No.1 pp.1-7, 2009.

Creep-Rupture Behavior of Die-Cast Magnesium Alloys

Yakov B. UNIGOVSKI

Department of Materials Engineering, Ben-Gurion University of the Negev,
84105, P.O.B. 653, Beer-Sheva, Israel

Received Feb. 13, 2009
Accepted Apr. 27, 2009

Abstract

It is found that the relationship between the time-to-rupture t_r and the time to the onset of tertiary creep t_{ot} (t_r/t_{ot}) in die-cast Mg-9Al alloy is equal to 1.3 and independent of stress, test temperature and die-casting parameters over the range examined. It is shown that the introduction of creep-rupture strain into Monkman-Grant relation modified by Dobeš and Milička demonstrates only the deviation range of the minimum creep rate from the average creep rate. The apparent activation energy E_c of creep is below the value of magnesium self-diffusion energy E_{SD} up to 175°C. At higher temperatures, $E_c > E_{SD}$, and creep deformation may be controlled by diffusion processes (dislocation climbing, etc.). The established magnitude of stress exponent n in the power law (the modified Arrhenius rate equation) within the stress range from 20 to 50 MPa is close to the value of $n \cong 3$ typical of solid-solution alloys. The increase in n values at higher stresses up to ~ 5 is caused, probably, by stress-induced precipitation of β-phase during long-term creep tests.

Key words : Die-Cast Mg alloys, Creep rupture, Monkman-grant relation, Apparent activation energy.

Introduction

The most widespread die-cast Mg-alloy AZ91D (Mg - 9% Al - 1% Zn) has a high castability and a relatively high corrosion resistance. On the other hand, automotive and aerospace applications of Mg-Al alloys are mainly limited by their insufficient creep resistance at elevated temperatures. For a designer, the most important parameters are the minimum creep rate (*MCR*) at the steady-state creep stage and time-to-rupture (t_r). Therefore, their dependence on temperature and stress are of special interest for the understanding of creep mechanisms. Various creep mechanisms and equations have been proposed, which can be subdivided into two basic groups: diffusional creep and dislocation creep.[6, 12, 2] In case of diffusional creep corresponding to relatively high temperatures above 0.6 of the melting point T_m (Kelvin scale), the diffusion of single atoms either by bulk transport (Nabarro-Herring creep) or by grain-boundary transport (Coble creep) leads to a Newtonian viscous flow without any motion of dislocations. However, for structural applications, the temperature range from about 0.4 to $0.6T_m$ is the most important.[17] For AZ91D alloy this range corresponds to temperatures varying approximately from 20°C to 170°C. Creep deformation in this regime can be associated with a combination of dislocation glide mechanism typical of $T < 0.4T_m$ and a mechanism involving additional slip systems operating at elevated temperatures. Dislocations acquire the ability to climb from one slip system to another through diffusional processes.[17]

The apparent creep activation energy E_c is determined according to the Norton power law (the modified Arrhenius rate equation):

$$MCR = A\sigma^n \exp(-E_c/RT), \text{ \%/h}$$

where A is constant; R is gas constant; σ is the applied stress, T is the absolute temperature.

For Mg-0.8%Al alloy (Vagarali and Langdon, 1982) and pure aluminum (Sherby and Burke, 1969), E_c value grows with test temperature increasing within the range of 120-200°C. For solid-solution alloys, the stress exponent n is close to the value of $n \cong 3$.[18, 13] However, literature data about n values for Mg-Al and Mg-Al-Zn alloys are very contradictory. For example, it has been documented that the stress exponent values for these alloys are observed in the range from 2 to 6.[20, 21, 11, 5, 16, 3] For die-cast AZ91D alloy in the temperature interval of 150-180°C and stress lower than 60 MPa, n changes from 2.0-2.2 (Dargush, *et al*. 1998) to 4.7.[4]

In the present paper we establish some relationships for creep-rupture behavior and find the apparent activation energy of creep for die-cast AZ91D Mg alloy in order to understand the creep mechanisms.

Materials and Experimental Procedures

Specimens of AZ91D alloy with a typical chemical composition, wt.%: ~9.0 Al; 0.2 Mn ; 0.7 Zn; 0.015 Si; 0.0004 Ni; 0.003 Fe; 0.0006 Be; Mg - the rest, produced on die-cast 40-t cold-chamber machine (Israeli Institute of Metals - Technion) and 500-t hot chamber machine (Ortal Diecastings Ltd, Israel) were 6 mm in diameter, with the gage length of 30 mm.

Creep tension tests with the duration of 20-400 hours were carried out on Model 3 Satec machine (Satec System, Inc., USA) at the test temperatures and stresses ranging from 120 ± 1°C to 200 ± 1°C and from 20 MPa to 75 MPa, respectively. All creep tests were carried out on 5-10 specimens for each set of casting conditions. Since creep flow rate decreases in the beginning of a creep test down to a minimal value and remains approximately constant for a long time afterwards, this minimum creep rate was accepted as a characteristic of creep resistance. In addition, the results of creep tests performed at room temperature in air and in borate buffer solution named earlier corrosion creep (Unigovski, *et al.* 2005) were used for the analysis of creep-rupture behavior of polycrystalline 99.9653% Mg and die-cast alloys AM50 (5.1% Al, 0.15% Zn, 0.57% Mn, Mg-balance) and AS21 (2.3% Al, 0.23% Mn, 1.10% Si, Mg-balance).

Typical mechanical properties of pure Mg, AZ91D, AM50 and AS21 alloys, respectively, were as follows: the UTS equal to 62, 225, 229 and 221 MPa, tensile yield strength (TYS) equal to 37, 170, 136 and 134 MPa, elongation-to-fracture equal to 5.7, 2.8, 11.7 and 9.7%.

Microstructure studies were carried out using an optical microscope "Nicon" with a computer program "Omnimet" for quantitative phase analysis at the magnification of 1:200 and a scanning electron microscope Jeol JSM-35CF with energy dispersive spectroscope "Link system" AN 10000.

Results and Discussion

Typical creep curves for AZ91D alloy at 150 and 175°C under a stress of 50 MPa are shown in Figure 1. Regions I, II and III in curve 2 characterize primary (transient), secondary (steady-state) and tertiary creep, respectively. As known, the primary creep is characterized by strain hardening and a decreasing creep rate. The steady-state region is characterized by a constant minimum creep rate. The duration of the primary creep t_I and creep resistance of the alloy decrease significantly with the specimen porosity growth. For example, with the porosity growth from 1.5 to 4.5%, t_I decreases from 120 to 25 hours. Temperature increase leads to a shortening of the primary creep duration and to a significant growth of the minimum creep rate. Thus, with temperature growth from 150°C to 175°C, t_{pr} is shortened from 120 to 25 hours. The results included into Figure 1. show a marked shortening in the strain hardening period (transient creep) of AZ91D alloy with the porosity and/or temperature growth. Dotted lines denoted as *ACR* and *MCR* represent the slope for calculating average and minimum creep rates, respectively, for curve 2. Here the average creep rate of the material in the creep test $ACR = \varepsilon_r / t_r$, where ε_r is the creep-rupture strain.

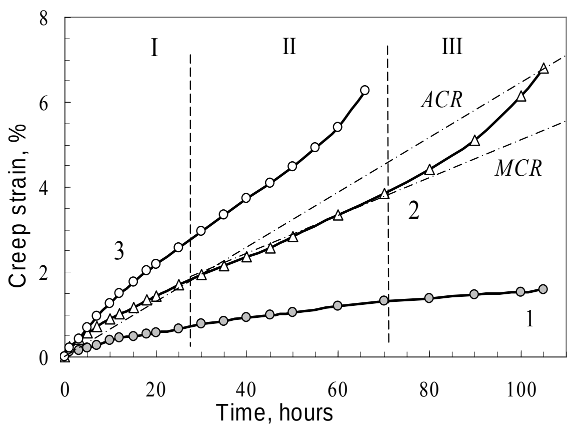


Figure 1. Typical creep curves of AZ91D alloy for specimens with low and high porosity *P*. *P*, %: ~1.3 (1, 3) and ~5 (2); test temperature, °C: 150 (1, 2) and 175 (3); σ = 50 MPa. Curve 2: I, II and III - areas of primary, secondary and tertiary creep, respectively. Dotted lines denoted as *ACR* and *MCR* show the slope for calculating average and minimum creep rates, respectively, for curve 2.

The relationship between the time-to-rupture t_r and the time to the onset of tertiary creep $t_{ot}=t_I + t_{II} = t_r - t_{III}$ (t_{II} and t_{III} are the durations of the second and third stages) for die-cast AZ91D produced on the 40-t machine is shown in Figure 2. with t_r/t_{ot} ratio ≅ 1.30 ($t_r = 1.3006\ t_{ot} + 14.74$; the correlation coefficient R = 0.95) independent of the stress σ, test temperature T and die-casting parameters over the range examined (casting temperature T_{cast} = 630-740°C; die temperature T_{die} = 180-235°C; injection rate V_g = 11-33 m/s; σ = 50÷75 MPa, T = 150÷175°C).

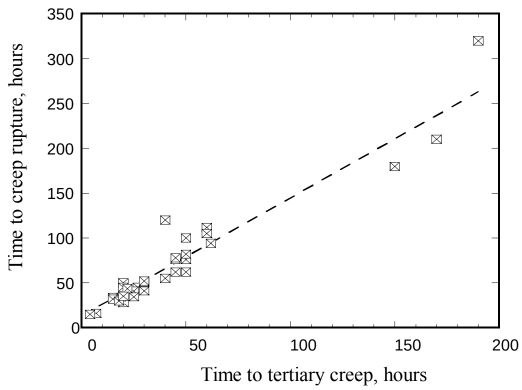
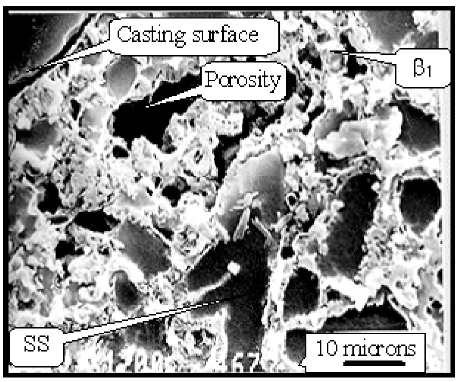
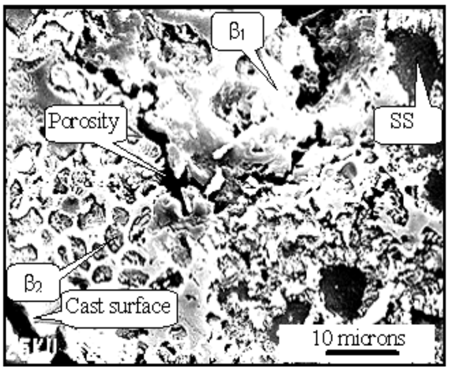


Figure 2. The time-to-rupture t_r vs. the time to the onset of tertiary creep t_{ot} for die-cast AZ91D.

AZ91D alloy is a structurally instable material during a relatively prolonged creep test at elevated temperatures. Its microstructure is altered due to the precipitation of fine intermetallic β–phase $Mg_{17}Al_{12}$, mainly, within the grains of Mg-Al-Zn solid solution. As-cast alloy contains relatively coarse eutectic intermetallic β_1 between the grains of solid solution (Figure 3a) and fine intermetallic β_2 precipitated during a long-term creep test (Figure 3b).



(a)



(b)

Figure 3. SEM micrographs showing the microstructure of die-cast AZ91D alloy in the vicinity of the surface layer before (a) and after (b) the creep-rupture test (150°C, 50 MPa, 400 hours). β_1–coarse eutectic intermetallic, SS – grains of Mg-Al-Zn solid solution; β_2–fine intermetallic precipitated during a long-term creep test.

The empirical log-log relationship between the time-to-rupture and minimum creep rate is known in the literature as Monkman-Grant (M-G) relation log t_r + m log MCR = C, where m and C are constants.[(14)] A more appropriate description is given by a relation suggested by Dobeš and Milička (further, D-M), which includes deformation-modified rupture time:

$$\log\frac{t_r}{\varepsilon_c} + m'\log MCR = C',$$

where m' and C' are constants; ε_c is the creep strain at fracture (Dobeš and Milička, 1976).

It can be easily seen that t_r/ε_c ratio in the D-M relation equals $(ACR)^{-1}$. Therefore, the second relation may be represented as m' log MCR – log $ACR = C'$. The Table presents the results of creep data fitting using these relationships for die-cast magnesium alloys (AZ91D, AM50 and AS21) and pure commercial Mg tested at room temperature (RT) in buffer solution (Unigovski, *et al.* 2005) and elevated temperatures (ET= 120°C ÷ 175°C) in air under stresses of σ = 16÷60 MPa. Obviously, the data for AZ91D alloy produced both in cold- and hot-chamber machines are well described by log ACR-log MCR relation with higher values of the correlation coefficient R and, of course, smaller scatter of ACR values in comparison with the scatter of time-to-rupture as functions of the minimum creep rate (Table, Figure 4a).

Table. 1. Fitting of the results using Monkman-Grant (1) and Dobeš-Milička (2) relations

No.	The alloy and test conditions	(1) $\log t_r + m \log MCR = C$			(2) $m' \log MCR - \log ACR = C'$		
		m	C	R	m'	C'	R
1	AZ91D, ET cold chamber	0.7919	0.7993	0.76	0.9573	-0.0818	0.97
2	AZ91D, ET, hot chamber	0.8442	0.7906	0.89	1.0247	-0.1550	0.93
3	Mg and all alloys, RT, ET	0.2771	1.4029	0.58	0.8510	-0.0353	0.99

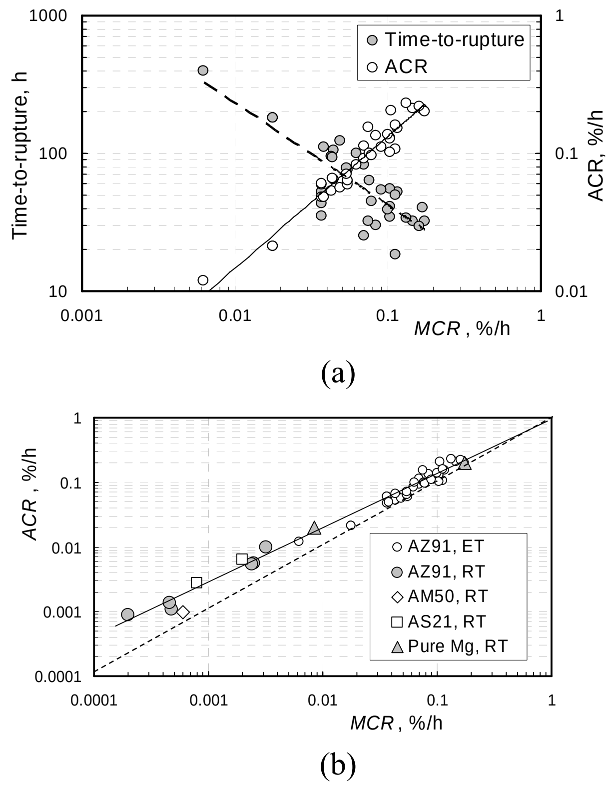


Figure 4. Time-to-rupture (t_r) and average creep rate ($ACR = \varepsilon_f / t_f$) as functions of minimum creep rate (MCR) for AZ91D alloy tested at elevated temperatures (a) and for a group of cast alloys and pure Mg tested both at room temperature (RT) in borate buffer solution and at elevated temperatures (ET = 120°C÷175°C) in air (b) under stresses of σ= 16÷60 MPa.

In general, the average creep rate *ACR* is always higher than *MCR* value (Figure 1) or close to it for brittle materials with relatively short durations of primary and tertiary stages t_I and t_{III} . Therefore, as it was to be expected, the data of more than 50 creep-rupture tests for all alloys and pure Mg tested at room and elevated temperatures and represented in log *ACR* – log *MCR* coordinates showed a very small scatter of the results with R = 0.99, especially at high values of *MCR* (close to 0.01 %/h and higher), when $MCR \approx ACR$ (Table, Figure 4b). Therefore, the coefficients m' and C' show only the deviation scope of *MCR* from the average creep rate. Of course, the fitting of these data obtained for all metals, including the results of corrosion creep at room temperature, in log t_r - log *MCR* coordinates showed a very low R value of 0.58 and an inadequacy of M-G relation for these conditions (see Table). Although M-D relation may improve the correlation, it should be taken into account that the introduction of creep elongation factor may be of value when only a correlation or interpolation of test results is desired, but it is not recommended for extrapolation.[22] Moreover, in some examples, a spread in creep elongation of type 304 stainless steel obtained using M-D relation exhibited from 2% to 24% at the same goodness of fit (R = 0.93) for M-G and D-M log-log relationships.[7]

According to the slope of straight-line portions of ln *MCR* - 1/*T* dependencies, at σ= 50 MPa the apparent creep activation energy amounted to 44 and 106 kJ/mole in the test temperature ranges of 120-135 and 135-175°C, respectively, remaining below the value of magnesium self-diffusion energy E_{SD}, which amounts to 134.6 kJ/mole.[1] For a lower load (20 MPa), the value of E_c in the ranges of 150-175 and 175-200°C amounted, respectively, to 50 and 167 kJ/mole (Figure 5). At the temperature above 175°C and the stress of 20 MPa, E_c exceeds the self-diffusion energy of magnesium; therefore, diffusion processes (dislocation climbing, etc.) may control the process of creep strain in these test conditions.

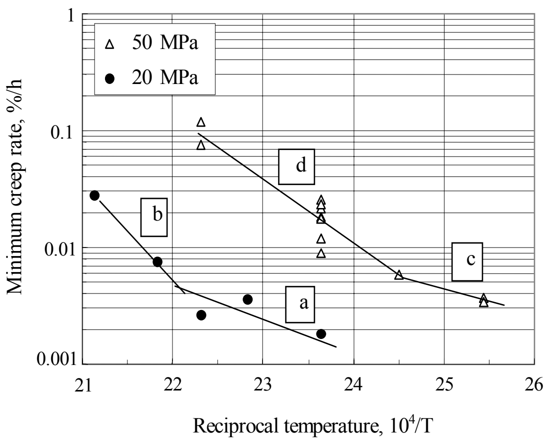


Figure 5. Minimum creep rate of AZ91D alloy vs. the reciprocal absolute temperature. The slope of straight-line sections corresponds to the apparent creep activation energy, kJ/mole: 50 (a), 167 (b), 44.2 (c) and 106 (d).

The apparent activation energy of creep grows with test temperature increase within the range of 120-200°C. This agrees with the data obtained earlier for Mg-0.8%Al alloy (Vagarali and Langdon, 1982) and pure aluminum.[18]

The established magnitude of the stress exponent $n = 2.2$ at 150°C within the range of σ from 20 to 50 MPa is close to the value of $n \cong 3$ for solid-solution alloys.[18, 13] At stresses above 50 MPa, it was found that $n \cong 5$. That corresponds to n value growth up to 5-6.9 observed earlier for die-cast AZ91D alloy in the temperature interval of 150-180°C and $\sigma >$ 50-60 MPa.[5, 16] The increase in n values at higher stresses is, probably, due to the additional stress-induced precipitation of β-phase during long-term creep tests. It is known (Williams and Wilshire, 1977, Jones, 1992 and Askeland, 1994) that for precipitation- and dispersion-strengthened alloys the reported values of n can range as high as 30 to 40 due to the interaction stresses between dislocations and barriers to the dislocation motion during creep.

The grain size of Mg-Al solid solution in the surface layer about 0.5 mm thick and in the bulk of die castings amounts to 3-10 microns and 10-15 microns, respectively. Quantitative phase analysis shows a weak dependence of $Mg_{17}Al_{12}$ intermetallic (β-phase) content on die-casting conditions.[8] In this work it was found that the volume fraction of β-phase located, as a rule, along grain boundaries, varies in the surface layer of about 0.5 mm thick (skin) within the limits of 25-32% for as-cast samples produced at different die-casting parameters. As shown in Figure 3, the volume fraction of β-phase grows, and the grain size of solid solution significantly decreased during tests at elevated temperatures. The volume fraction of β-phase in the surface layer 0.5 mm thick grows with increasing creep test duration from the initial value of 28% to 42% after 400 hours (150°C, 50 MPa) in samples produced under the same casting conditions. Under a synergistic action of stress and temperature, β-phase precipitates in the form of fine precipitates β_2 on grain boundaries (Figure 3).

Literature data regarding the influence of β-phase content on the mechanical properties of Mg alloys are also contradictory. It is supposed that the β-phase, which is relatively soft at elevated temperatures, allows for easy creep along the grain boundaries.[15] However, discontinuous precipitates of β-phase improve creep resistance of permanent-mold-cast AZ91D alloy (Gutman, *et al.* 1997), but they do not affect creep and stress relaxation characteristics of die-cast AZ91D alloy.[9]

On the other hand, at larger magnifications in TEM micrographs of die-cast AZ91D alloy, fine continuous precipitates of β-phase were also observed in grains of the solid solution.[5] Apparently, strain hardening of AZ91D alloy is related both to dispersion hardening of solid solution by fine continuous precipitates formed in the grains (Blum, *et al.* 1998) and to the formation of other obstacles impeding dislocation motion. Work softening of die-cast alloys after achieving the maximum creep resistance is related to the development of cavitation as a process of growth, merging and propagation of cavities.

Conclusions

1. It is shown that coefficients m' and C' in Monkman-Grant relation modified by Dobeš and Milička show only the deviation range of the minimum creep rate from the average creep rate. Although this relation improves the goodness of fitting, it should be taken into account that the introduction of creep rupture strain may be of value when only a correlation or interpolation of test results is desired.

2. The relationship t_r/t_{ot} between the time-to-rupture t_r and the time to the onset of tertiary creep t_{ot} is equal to 1.3 and independent of stress, test temperature and die-casting parameters over the range examined.

3. At stresses less than 50MPa, the apparent activation energy E_c of creep for AZ91 alloy is below the value of magnesium self-diffusion energy up to the test temperature of 175°C. At the temperature above 175°C, E_c exceeds the self-diffusion energy of magnesium and the process of creep strain may be controlled by diffusion processes (dislocation climbing, etc.).

4. The established magnitude of stress exponent n within the range of stresses σ from 20 to 50 MPa is close to the value of $n \cong 3$ typical of solid-solution alloys. The increase in n values at higher stresses up to ~ 5 is caused, probably, by stress-induced precipitation of β-phase during long-term creep tests.

Acknowledgments

The author is deeply grateful to Prof. E.M. Gutman (Ben-Gurion University of the Negev) for his kind help and useful discussion of the results and to Dr. Z. Koren and Mr. H. Rosenson (Israel Institute of Metals, Technion. Haifa) for manufacturing of die-cast specimens.

References

1. Askeland, D.R. 1994. *The science and engineering* of Materials. 3rd ed. Boston : PWS Publishing : 819.

2. Bernasconi, G. and Piatti, G. 1979. *Creep of Engineering Materials and Structures.* London : Applied Science Publishers**.**

3. Blum, W., Watzinger, B. and Weidinger, P. 1998. Creep resistance of Mg-base alloys. In: *Mordike, B.L. and Kainer, K.U. (eds.), Magnesium Alloys and their Applications, MATINFO Werkstoff-Informationgeselschaft*. Frankfurt, Germany : 49-60.

4. Chen, F.C., Jones, J. W., Mc Ginn, T.A., Kearns, J.E., Nielsen, A.J. and Allison, J.E. 1997. Bolt-load-retention and creep of die-cast Mg-alloys, In : *Proc. Int. Congress & Exposition, Detroit, Michigan, February 24-27, 1997, SAE technical paper series, Paper Number 970325*. Warrendale: The Society of Automotive Engineers. Pa : 13-21.

5. Dargush, M., Hisa, M., Caceres, C.H., and Dunlop, G.L. 1996. Elevated temperature deformation of die cast Mg alloy AZ91D. In : *Proceed. Third Int. Magnesium Conf.* The Institute of Materials, Manchester, England : 153-165.

6. Gittus, J. 1975. *Creep, viscoelasticity and creep fracture in solids*. London : Applied Science Publishers : 725.

7. Gold, M., Leyda, W.E. and Zeisloft, R.H. 1975. The effect of varying degree of cold work on the stress-rupture properties of type 304 stainless steel. *Trans. ASME J. Eng. Mat. Technol*. **97(4) Series H** : 305–312.

8. Gutman, E.M., Unigovski, Ya.B., Levkovich, M., Aghion, E. and Dangur, M. 1997. Influence of technological parameters of permanent mold casting and die casting on creep and strength of magnesium alloy AZ91D. *Mat. Sci. Eng. A*, **A234-236** : 880-883.

9. Gutman, E.M., Unigovski, Ya.B., Levkovich M. and Koren, Z. 1998. Porosity and casting condition influence on creep of die cast Mg-alloy. *J. Mater. Sci. Lett*. **17** : 1787-1789.

10. Jones, W. 1992. Theory of creep deformation. In : *ASM Metals Handbook*. 10th ed. Vol. 8, Mechanical Testing. New York : ASM International, the Material Information Society : 308-310.

11. Miller, W.K. 1991. Creep of die-cast AZ91D magnesium at room temperature and low stress. *Metall. Trans. A*. **22(A)** : 873-877.

12. Mitra, S.K. and McLean, D. 1966. Work Hardening and Recovery in Creep. *Proc. R. Soc.* **295** : 288–299.

13. Mohamed, F.A. and Langdon, T.G. 1994. The transition from dislocation climb to viscous glide in creep of solid solution alloys. *Acta Metall*. **22** : 779-788.

14. Monkman, F.C. and Grant, N.J. 1956. An empirical relationship between rupture life and minimum creep rate in creep-rupture tests. *Am. Soc. Test. Mater. Proc*. **56** : 593-620.

15. Raynor, G.V. 1959. *The physical metallurgy of Magnesium and its Alloys*. London : Pergamon Press : 521.

16. Regev, M., Aghion, E. and Rosen, A. 1997. Creep studies of AZ91D pressure die-casting. *Mater. Sci. Eng. A*. **A234-236** : 123-126.

17. Sherby, O.D. 1962. Factors affecting the high temperature strength of polcyrystalline solids. *Acta Metal*. **10** : 135–147.

18. Sherby, O.D. and Burke, P.M. 1969. Mechanical behavior of crystalline solids at elevated temperature. *Prog. Mat. Sci*. **13** : 325-390.

19. Unigovski, Ya., Keren, Z. Eliezer, A. and Gutman, E.M. 2005. Creep behavior of pure magnesium and Mg–Al alloys in active environments. *Mater. Sci. Eng.* **A398(1-2)** : 188-197.

20. Vagarali, S.S. and Langdon, T.G. 1981. Deformation mechanism in H.C.P. metals at elevated temperatures-I. Creep behavior of magnesium. *Acta Metall.* **29** : 1969-1982.

21. Vagarali, S.S. and Langdon, T.G. 1982. Deformation mechanism in H.C.P. metals at elevated temperatures-II.Creep behavior of a Mg-0.8% Al solid solution alloy. *Acta Metall.* **30** : 1157-1170.

22. Voorhees, H.R. 1992. Assessment and Use of Creep-Rupture Properties. In : *ASM Metals Handbook*. 10^{th} ed. Vol. 8, Mechanical Testing. New York : ASM International, the Material Information Society : 838-885.