

Effect of Die-Casting Conditions on Viscoelastic Behavior of Mg Alloy

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Creep and stress relaxation resistance of the most common die-cast Mg alloy AZ91D (Mg-9% Al-1% Zn) are influenced by both casting temperature and injection rate as well as by die temperature and porosity. Relationships between viscoelastic properties of Mg alloy at 150 °C and the parameters of die-casting technology are obtained and presented as simple contour plots (isograms). These properties can be improved by reducing casting temperature and increasing injection rate. When fabricating complicated thin-wall components, castability of the alloy can be diminished due to a fast temperature drop. In this case, casting temperature should be higher, whereas injection rate can be reduced. Porosity seems to be the most important characteristic of die-cast alloy microstructure affecting its viscoelastic properties. Coarse β-phase precipitates do not affect creep and relaxation characteristics of die-cast AZ91D alloy.

Keywords magnesium alloys, creep, stress relaxation, die-casting parameters, porosity

1. Introduction

The most efficient method of Mg alloys casting is pressure die casting. The most widespread Mg alloy AZ91D (Mg-9% Al-1% Zn) has high castability and relatively high corrosion resistance. On the other hand, automotive and aerospace applications of this alloy are mainly limited by its insufficient creep resistance and stress relaxation characteristics at elevated temperatures. For example, an increase in working temperatures in a modernized engine can result in stress relaxation under bolts, loss of clamp load, and oil leakage.[1,2] Mg-Al-Si (AS41, AS21) and Mg-Al-RE (AE42) alloys have considerably higher creep resistance in comparison to Mg-Al-Zn-Mn alloys. However, these alloys have some drawbacks. Thus, for instance, the salt corrosion rate of die-cast AS21 and AS41 alloys is 1.5 to 3 times higher than that of AZ91D and AE42.[2,3,4] Meanwhile, the castability of AE42 alloy is significantly lower, and its cost is considerably higher than that of AZ91D alloy.[2]

The limits of possible improvement of mechanical properties of Mg-Al-Zn-Mn alloy system are presently considered unattained. The exploration of ways of improving the properties (primarily, creep and stress relaxation behavior) of Mg alloys by optimizing die-casting process parameters is of considerable scientific and practical interest. Meanwhile, literature data on the dependence of mechanical properties of Mg alloys on casting parameters are rather contradictory even for standard tensile properties.[5,6,7] As for stress relaxation, such data are practically nonexistent. In the present paper, we establish correlation relations for the impact of porosity and die-casting parameters on creep and stress relaxation resistance of the Mg alloy AZ91D in order to optimize the quality of cast components.

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2. Experimental Procedures

The specimens of AZ91D alloy (wt.%:9.3Al, 0.21Mn, 0.71Zn, 0.015Si, 0.0004Ni, 0.0028Fe, 0.0006Be, and Mg—balance) produced on a die-cast cold-chamber machine with the locking force of 400 kN were 6 mm in diameter, with gage length in creep and stress relaxation tests of 30 and 75 mm, respectively. Casting parameters were varied within the following ranges: casting temperature $t_{cast} = 630$ to 740 °C, die temperature $t_{die} = 180$ to 235 °C, and injection rate $V_g = 11$ to 33 m/s. These ranges of casting parameters were characteristics of this die-casting machine.

Creep tension tests with the duration of 20 to 400 h were carried out on a model 3 Satec (Satec System, Inc., USA) machine at 150 ± 1 °C. All creep tests were carried out on five to ten 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 afterward, this minimum creep rate (MCR) was accepted as a characteristic of creep resistance.

Stress relaxation tension tests with the duration of 22 h were performed on a Zwick-1445 (Zwick GmbH & Co., Germany) machine at 150 ± 1 °C. The initial stress in stress relaxation tests was equal to 67 MPa, or 70% of tensile yield strength. The final value of stress (here, after 22 h) or the remaining stress σ_{rem} was accepted as a characteristic of relaxation resistance of the alloy. It is well known [8] that relaxation behavior of a material is characterized by a spectrum of relaxation times or a relaxation spectrum $H(\tau)$. In the first approximation,

$$H(\tau) = -\left[dE(t) / d\ln t\right]_{t-\tau}$$

where $E(t) = \sigma(t)/\varepsilon_0$ is the relaxation modulus, t is the current time, $\sigma(t)$ is the current relaxation stress, ε_0 = constant is the initial (elastic) strain, and τ is the relaxation time. The function $H(\tau)$ also was accepted as a characteristic of stress relaxation.

The specimen density was determined according to Archimedes principle. The absence of multiple correlations between P and controllable casting parameters (t_{cast}, t_{die}, and V_g) allows us to consider the porosity of castings as a variable, along with controllable parameters.[9] 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 (SEM) Jeol JSM-35CF (Japan Electron Optics Ltd., Tokyo) with an energy dispersive spectroscope "Link system" AN 10000.

3. Results and Discussion

Figure 1 shows the relaxation modulus $E(t)$ and relaxation spectrum $H(\tau)$ plotted against t and τ for a typical specimen. It is found that the relaxation time τ_o corresponding to the maximum of function $H(\tau)$ depends on casting parameters and porosity. The relaxation time usually varies from 10 to 20 h. Note that the relaxation spectrum is only a mathematical description of macroscopic behavior, which does not necessarily have a simple interpretation at the molecular level.

Correlation analysis of the results of 24 stress relaxation tests and 45 creep tests was performed. We obtained nonlinear correlation equations connecting such parameters as casting temperature, injection rate, and porosity with minimum creep rate, relaxation remaining stress, and relaxation time. The coefficients of the multiple correlation R were equal to 0.72 to 0.80. We have established that the effect of the variable t_{die} varying within the range of 180 to 235 °C on viscoelastic properties of the alloy is insignificant in comparison with other variables.

For example, MCR depends on casting temperature, injection rate, and porosity as follows ($R = 0.80$):

$$(MCR) \times 10^4, \%/\mathrm{h} = 2463 + 1.246\, t_{cast} - 295.38\, V_g + 378.91\, P - 0.00871 (t_{cast})^2 + 0.0158 (V_g)^2 + 0.71 P^2 + 0.4766\, t_{cast}\, V_g - 0.323\, t_{cast}\, P - 3.154\, V_g\, P$$

which is valid at $t_{cast} = 630$ to $740\,°\mathrm{C}$, $V_g = 11$ to 33 m/s, $t_{die} = 230\,°\mathrm{C}$, and $P = 0.5$ to 7%.

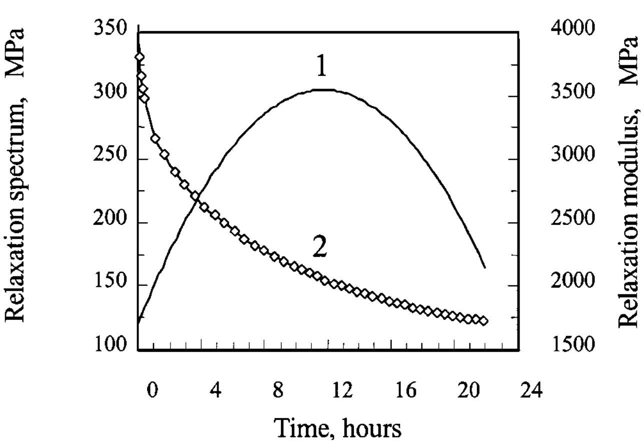


Fig. 1 Relaxation spectrum (1) and relaxation modulus (2) vs time for a typical specimen ($t_{cast} = 687$ °C, $V_g = 22$ m/s, $t_{die} = 230$ °C, and $P = 2.6\%$)

Contour plots, or isograms (Figs. 2 to 4) generated by nonlinear equations demonstrate the influence of parameters of die-casting process (injection rate and casting temperature) on viscoelastic properties of the alloy at specified values of porosity and die casting temperature ($P = 2\%$, and $t_{die} = 230$ °C). The injection rate increase from 13 to 30 m/s halves the relaxation time, but the latter is almost independent of casting temperature (Fig. 2). Figure 3 shows that the remaining stress values exceeding 60% (contour lines 5 to 8) are observed at a reduced casting temperature (<637 °C) and an increased injection rate (>23 m/s) or at an increased casting temperature (>685 °C) and a reduced injection rate (<19 m/s). Creep resistance of the alloy depends on die-casting parameters similarly to stress relaxation dependencies. Minimum creep rate lower than 0.020%/h is observed at $t_{cast} < 630$ °C and $V_g > 27$ m/s or at $t_{cast} > 647$ °C and $V_g < 22$ m/s (Fig. 4, contour lines 1 to 6).

The estimation of hydrodynamic behavior of molten metal and thermal situation in a die cavity as a function of injection rate shows the following. During the cavity filling, we can distinguish, depending of V_g value, laminar, continuous turbulent, and dispersed turbulent metal flow rates.[10] Injection rate values for a laminar flow and continuous turbulent flow should not exceed certain critical values V_1 and V_2, respectively. At the injection of Mg alloys in the liquid state and at temperatures close to the liquidus temperature, the values of V_1 amount to 0.5 to 0.6 and 2 to 5 m/s, respectively. Here, $V_2 = cv^m\,(bg/b)^n$, where v is the kinematic viscosity, m²/s; b and b_g are casting wall and gate thicknesses; and c, m, and n are coefficients equal to 75, 0.52, and 0.65, respectively, for Mg alloys. In our case, at the gate thickness of 2 mm and the specimen diameter of 6 mm ($b_g/b = 0.33$), $V_2 = 45 \pm 3$ m/s is somewhat different from 31 ± 2 and 24 ± 2 m/s reported for aluminum and zinc alloys, respectively.[10] Thus, casting conditions in our experiments correspond to continuous turbulent metal flow rates in the die cavity. Under the condition of a continuous turbulent flow rate conservation $V_1 < V_g < V_2$, eddy motion at the boundaries of turbulent flows can lead to air entrainment and increase the porosity of castings. An increase in alloy properties at V_g growth

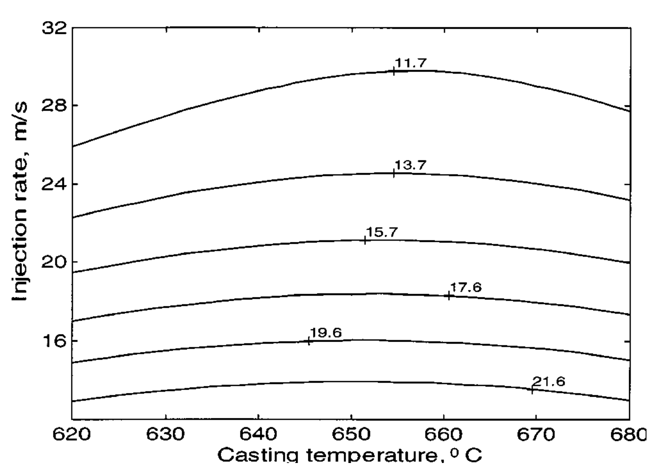


Fig. 2 Effect of die-casting parameters on the relaxation time (line numbers refer to hours) of AZ91D alloy at test temperature of 150 °C and the initial stress of 67 MPa ($t_{die} = 230$ °C, and $P = 2\%$)

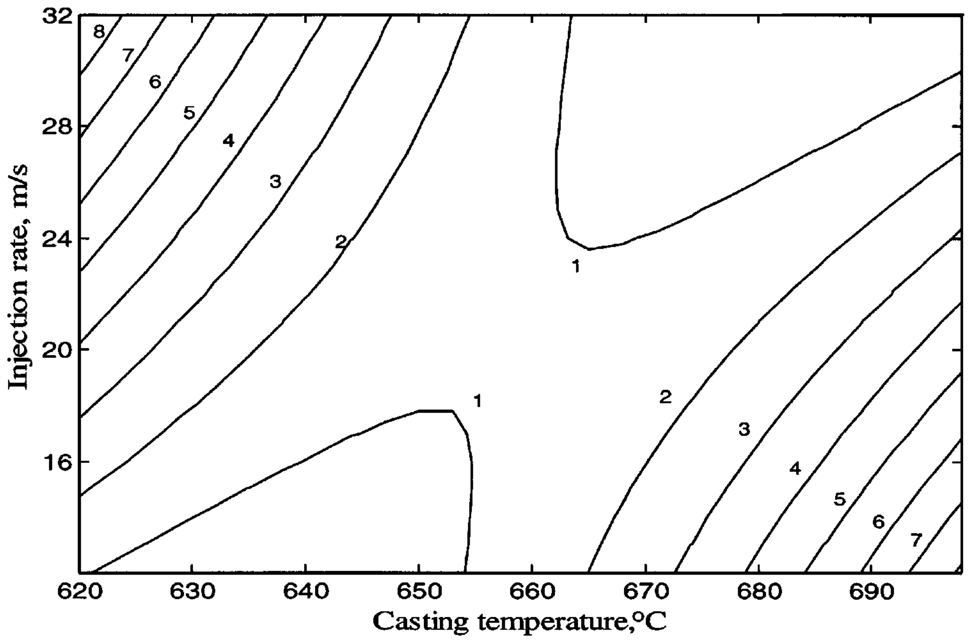


Fig. 3 Effect of die-casting parameters on remaining stress (σ_{rem}) of AZ91D alloy at the test temperature of 150 °C and stress of 50 MPa (t_{die} = 230 °C, and P = 2%). σ_{rem}, %: 48 (1), 52 (2), 56 (3), 60 (4), 64 (5), 67 (6), 71 (7), and 75 (8)

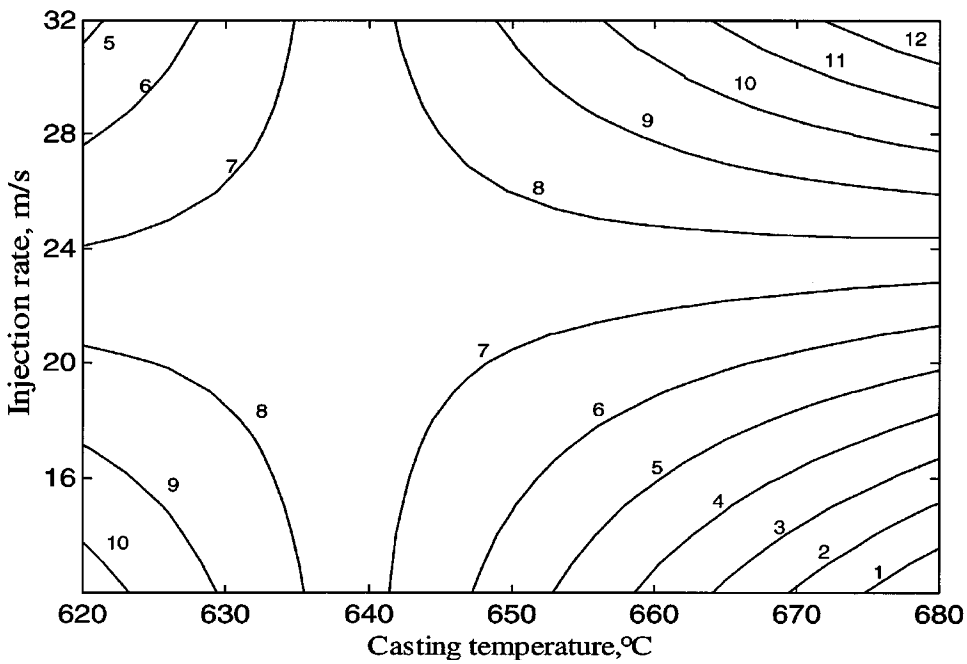


Fig. 4 Effect of die-casting parameters on *MCR* of AZ91D alloy at the test temperature of 150 °C and stress of 50 MPa (t_{die} = 230 °C, and P = 2%). MCR × 10^3, %/h: 4 (1), 7 (2), 10 (3), 13 (4), 16 (5), 19 (6), 22 (7), 25 (8), 28 (9), 32 (10), 34 (11), and 37 (12)

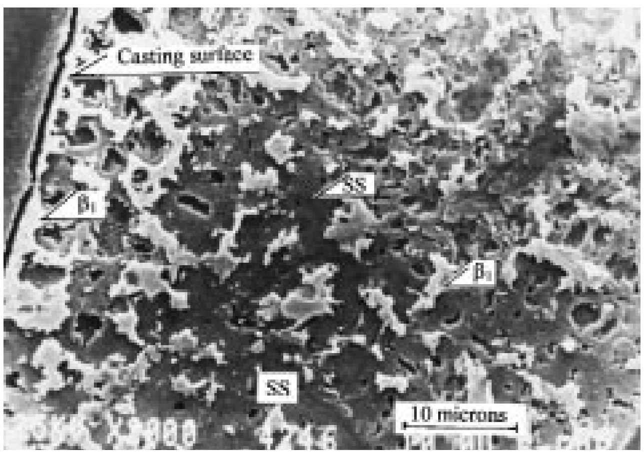
from lower limits up to certain values is, apparently, connected with metal density growth due to increase of the metal pressure. At higher injection rates, alloy density decreases,[11,12] which is connected, first, with air entrainment by dispersed metal flows during die filling and, second, with fast air expulsion out of die cavity through vents.

The injection rate growth up to the upper limit does not affect the thermal situation in the die cavity, for example, melt temperature. Its growth (Δt gradient) due to the transition of jet kinetic energy into heat at its breaking against the die wall can be estimated in the first approximation as follows: $mC\Delta t = m\,(V_g)^2/2$ or $\Delta t = (V_g)^2/2C$, where m is the mass of metal in the die cavity, and $C = 1420$ J/Kg K[13] is the heat capacity of the molten AZ91D alloy. Then Δt is equal to 0.9 and 3.5 K for the injection rates of 50 and 100 m/s, respectively.

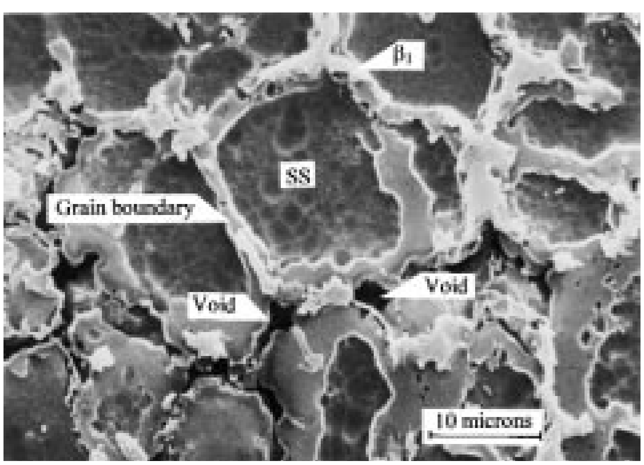
Casting temperature affects gas content in the molten alloy. Therefore, to decrease the alloy porosity[5,9] and to improve viscoelastic properties, one should choose a reduced casting temperature 630 to 640 °C and an elevated injection rate (Fig. 3 and 4). When fabricating complicated thin to wall articles, castability of the alloy can be diminished because of a fast temperature drop. In this case, casting temperature should be higher, whereas the injection rate can be reduced. The obtained dependencies of viscoelastic properties on die-casting parameters are of general nature and, apparently, can be extended to other mechanical properties. Thus, the highest values of the impact energy of Mg alloy AM50 (Mg-5%Al-0.4%Mn) produced on a die-casting machine with a locking force of 2000 kN can also be obtained at a reduced level of casting temperature (630 to 650 °C) and maximum V_g (65 to 90 m/s) or at t_{cast} exceeding 660 °C and V_g below 50 m/s.[14]

The grain size of Mg-Al solid solution (SS) in the surface layer about 0.5 mm thick and in the bulk of die castings amounts to 1 to 10 and 10 to 15 μm, respectively. Quantitative phase analysis shows a weak dependence of the $Mg_{17}Al_{12}$ intermetallics (β phase) content on die-casting conditions. It was found earlier[7] that the volume fraction of β phase located, as a rule, along grain boundaries varies in "as-cast" samples produced at different die-casting parameters within the limits of 25 to 32%. The increase in casting temperature within the range of 630 to 740 °C leads to a certain increase in the dimensions of the grain size of Mg-Al-Zn SS in the surface layer. As shown in Fig. 5 and 6, the volume fraction of β phase grew, and the grain size of SS significantly decreased during the tests at elevated temperatures both close to the casting surface (Fig. 5a and 6a) and in the center of the specimen (Fig. 5b and 6b). 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 h (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 (Fig.6a and b).

Literature data referring to the influence of β-phase content on the mechanical properties of Mg alloys are also contradictory.

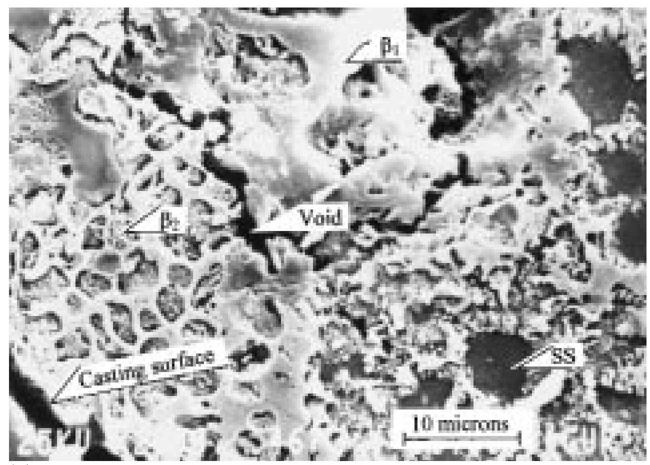


(a)

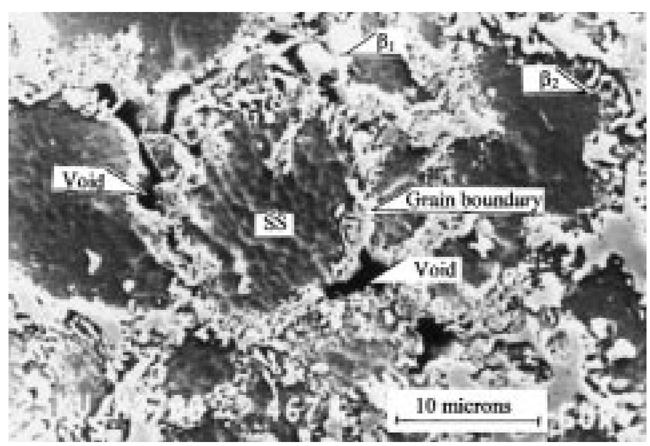


(b)

Fig. 5 Electron micrographs showing the microstructure of die-cast AZ91D alloy **(a)** in the vicinity of the surface layer and **(b)** in the center of as-cast specimen. β_1 are coarse eutectic intermetallics, and SS are grains of Mg-Al-Zn solid solution.



(a)



(b)

Fig. 6 Electron micrographs showing the microstructure of die-cast AZ91D alloy **(a)** in the vicinity of the surface layer and **(b)** in the center of specimen after a creep-rupture test. β_1 are coarse eutectic intermetallics, β_2 are finer intermetallics precipitated during a long-term creep test (150 °C, 50 MPa, 400 h), and SS are grains of Mg-Al-Zn solid solution.

It is supposed that the β phase, which is relatively soft at elevated temperatures, allows for easy creep along the grain boundaries.[16] However, discontinuous precipitates of β phase improve creep resistance of permanent-mold-cast AZ91D alloy,[7] but they do not affect creep and stress relaxation characteristics of die-cast AZ91D alloy.[18,19] On the other hand, at larger magnifications in transmission electron micrographs of die-cast AZ91D alloy, fine continuous precipitates of β phase were also observed in grains of SS.[15] Apparently, strain hardening of AZ91D alloy is related both to dispersion hardening of SS by fine continuous precipitates formed in the grains[15,17] 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.

4. Conclusions

The relations obtained in the present work show that the reduced casting temperature 630 to 650 °C and the increased injection rate improve viscoelastic properties. When fabricating complicated thin-wall components, the castability of the alloy can be diminished due to a fast temperature drop. In this case, casting temperature should be higher (660 to 690 °C, *etc.*), whereas injection rate can be reduced. In any case, the upper and lower injection rate limits are defined by a set of conditions intended to obtain high-quality components.

Porosity seems to be the most important characteristic of die-cast alloy microstructures, affecting their viscoelastic properties. Coarse β-phase precipitates do not affect creep and relaxation characteristics of die-cast AZ91D alloy. It is supposed that strain hardening of the alloy is caused both by the formation of fine precipitates of β phase and by other obstacles impeding dislocation motion in grains of Mg-Al-Zn solid solution. Work softening of the alloy after reaching maximum creep resistance is due to the cavitation process.

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