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Viscosity of Industrially Important Al–Zn Alloys. I-Quasi-eutectic Alloys

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Abstract Viscosity is a very important property in several fields of materials and metal processing. Many systems are still not fully characterized with regard to their thermophysical property data. In addition, as new alloys are developed or used, viscosity data are necessary to understand the best processing conditions. The data that are available frequently show large discrepancies. The effect of minor constituents is very difficult to model and can have a strong effect on the viscosity. Measurements of the viscosity of Zn–Al alloys, with a quasi-eutectic composition, were performed in the molten state, for temperatures between 690 K and 751 K. The measurements were performed with an oscillating cup viscometer, with an estimated uncertainty of 2 % to 5 %, depending on the alloy. The influence of other minor components, such as Pb, Mn, and Mg, on the viscosity of the alloys is discussed. It was concluded that Pb induces a decrease in viscosity, with Mg having the opposite effect, while the effect of Mn is not significant. All the alloys showed Newtonian behavior in the temperature range studied and a non-Arrhenius temperature dependence of viscosity, as usually found for pure molten metals.

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1 Introduction

High-temperature melts are very important in many industrial processes, such as steelmaking, non-ferrous metallurgy, aluminum smelting, foundries, glass manufacturing, etc. [1]. Viscosity, density, heat capacity, and thermal conductivity are by far the most important physical properties for fluid flow and heat transfer processes, and their values influence directly the optimal technological design of items in process plants or solar energy power stations, such as those of molten salts [2]. In fluid flow behavior, the kinetics of metallurgical and die casting processes, the viscosity and its reciprocal, the fluidity, are fundamental parameters.

Despite this fact, industry still needs new data for several pure metals and alloys, as the available data frequently show large discrepancies, and the effect of "impurities" is difficult to model. This may be attributed to technical difficulties in measuring the viscosity at high temperatures, a task that is both challenging for the experimentalist (and time consuming) and for theoretical interpretations. The need of accurate and reliable data for the viscosity of pure molten metals was recognized by the International Union for Pure and Applied Chemistry (IUPAC), and reference data for the viscosity of liquid aluminum and liquid iron were recently proposed [3]. Moreover, the existing methods of viscosity measurement and the metrology of this property, even at room temperature, have several problems, as recently discussed by Nieto de Castro et al. [4].

Within the several methods available for measuring the viscosity, those suitable for molten metals and alloys are [1,5]: (a) the capillary method, (b) the oscillating-body (cup, disk) methods, and (c) the rotational method. The most extensively used for liquid metals is the oscillating-cup method, as recently reported for Cu–Sn alloys [6], Ni-based alloys [7], and In–Se–Tl alloys [8]. Details of this method and working equations can be found elsewhere [9] and therefore will be only briefly presented here.

Any type of oscillating body viscometer consists of an axially symmetric body suspended from an elastic strand, where we can induce torsional oscillations. The viscosity of the fluid is obtained from the measured period and decrement of the oscillations. Once an oscillation is induced, the fluid will cause a damping in the oscillation, measured by the logarithmic decrement of its amplitude, and an increase in the oscillation period. These parameters depend on the viscosity and on the density of the fluid. The only measurements that are necessary, besides the state variables pressure and temperature, are mass and time, which can be obtained with high accuracy.

This is, in principle, an absolute method, as it is not necessary to perform any calibration with fluids of known viscosity. However, in addition to several details of the oscillating system that have to be rendered negligible during design, the measurement of temperature in the cup is quite tricky, causing additional uncertainty that has to be accounted for, namely, the calibration of the temperature measured by the thermocouple outside the oscillating couple and the "real" value inside the cup during the measurement. Therefore, we prefer to consider this method as a "quasi-primary method," in a metrological sense, which will become primary when all these experimental problems that degrade the uncertainty can be overcome [4].

The use of oscillating-body viscometers was restricted in the past due to the complexity of the mathematical treatment of the equations for the motion of the body. However, Kestin and Newell [10] and Beckwitt and Newell [11] provided solutions for these equations, triggering its extensive utilization, namely, for high-temperature melts. The viscosity calculation is obtained from the imaginary part of a rigorous solution for the oscillating-body viscometers, derived by Kestin and Newell [10]. This equation is

$$(s + \Delta_0)^2 + 1 + D(s) = 0 \tag{1}$$

where $D(s)$ is a characteristic function and s is the complex frequency of oscillation given by

$$s = \frac{T_0}{T}(-\Delta \pm \mathrm{i}) \tag{2}$$

Δ and Δ_0 are the logarithmic decrements of oscillation with and without fluid in the cell, and T and T_0 are the period of oscillation of the oscillating body with fluid and in vacuum, respectively. The characteristic function can be an exact solution [10] or an approximate solution derived by Beckwitt and Newell [11],

$$D(s) = s^2 \frac{I'}{I} \left(\begin{array}{l} \frac{4}{s^{1/2}\zeta_0} - \frac{6}{s\zeta_0^2} + \frac{3}{2s^{3/2}\zeta_0^3} + \frac{3}{2s^2\zeta_0^4} + \frac{1}{s^{1/2}z_0} \\ -\frac{16}{\pi\zeta_0 z_0} - \frac{9}{s^{3/2}\zeta_0 z_0} - \frac{8}{s^2\zeta_0 z_0} - \frac{\exp(-2s^{1/2}z_0)}{2s^{1/2}z_0} \end{array} \right) \tag{3}$$

where $\zeta_0 = R/(\eta T_0/(2\pi\rho))^{1/2}$ and $z_0 = h/(\eta T_0/(2\pi\rho))^{1/2}$ are dimensionless quantities for the inner radius of the cup, R, and the height of the fluid in the cup, h. I is the moment of inertia of the suspension system alone, I' is the moment of inertia of the fluid, η is the viscosity, and ρ is the density of the fluid. Equation 3 is applicable when $\zeta_0 \gg 1$ and $z_0 \gg 1$. From this general solution the working equation was derived for the oscillating-cup viscometer [10]:

$$\frac{\pi\rho h R^4}{2I}\left[A(p - \Delta q)\, x^{-1} - Bx^{-2} + Cqx^{-3}\right] = 2\left(\Delta - \frac{\Delta_0}{\theta}\right) \tag{4}$$

where

$$A = 4 + \frac{R}{h}$$
$$B = 6 + \frac{16R}{\pi h}$$
$$C = 1.5 + 9\frac{R}{h}$$
$$D = 1.5 - \frac{8R}{\pi h}$$

$$\begin{aligned}\theta &= \frac{T_0}{T}\\ p &= \left\{2\left[\Delta + \left(1+\Delta^2\right)^{1/2}\right]\right\}^{-1/2}\\ q &= \frac{1}{2p}\\ x &= R\left(\frac{2\pi\rho}{\eta T}\right)^{1/2}\end{aligned} \tag{5}$$

Equations 4 and 5 are used to calculate the viscosity from the period ratio and the logarithmic decrement. The equations are solved for the viscosity, giving

$$\eta = \frac{2\pi\rho}{T}\left(\frac{R}{x}\right)^2 \tag{6}$$

The error of this solution is smaller than 0.1 % when $R/h < 1$ and $x > 10$ [12]. In Eq. 3, the fluid equations used are the linearized Navier–Stokes equations for an incompressible fluid and are strictly applicable when the motion of the fluid is unidimensional, which we assume to be the case for the present instrument. Also, no effect of the vapor pressure above the liquid was considered, assuming that its effect on the overall torque induced in the mechanical system is negligible. Equation 6 was solved to obtain the viscosity of the liquid by an iterative method using a simple computer program in Basic language.

2 Experimental Apparatus and Method

The oscillating-cup viscometer is composed of four fundamental systems: an oscillating system (including the suspension system and oscillation initiator), a heating system, a vacuum system, and a system for the detection of oscillations. Figure 1 shows a schematic view of the measuring system, developed to measure the viscosity of molten materials, up to 1000 K, and previously described [9]. Figure 2 shows the viscometer assembly. This instrument has been previously used for measurements of the viscosity of molten salts [13,14]. The cylindrical cups used were made from 316 stainless steel. Samples of the alloys were received in the form of cylindrical rods (about 16.5 mm in diameter and 75 mm long), with the compositions given in Table 1. In the absence of phase diagrams for the multicomponent alloys, it was necessary to observe the melting of all the samples, prior to the viscosity measurements, and use the information available for the Zn–Al alloy, displayed in Fig. 3. The composition of the alloys (4.5 % or 5 % Al) is very close to the eutectic point ($T_{\mathrm{eu}} \sim 655$ K, $x_{\mathrm{Al,Eu}} \sim 6$ %).

The samples were produced by the Umicore group (http://www.umicore.com/en/), and their composition as received is displayed in Table 1. The main component is zinc (not shown), Al was in all the reported alloys at 5 % (mass percent) or 4.5 % (Alloy VIII), and the minor components are Mg, Pb, Si, and Fe (in all), and Mn (V and VI).

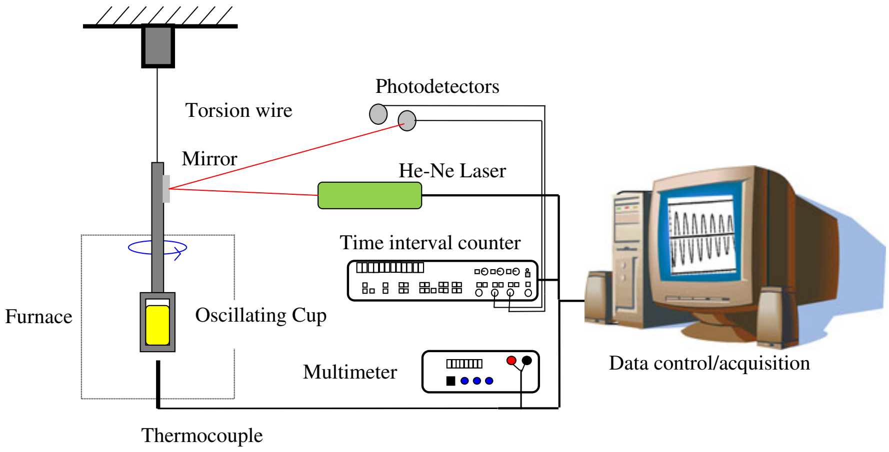


Fig. 1 Measuring system (schematic). Adapted from [2]

A sample of each of the alloys was pre-melted in porcelain crucibles (Haldenwager, Berlin, 79-2) in an oven to visualize any behavior different from an expected Newtonian liquid or dissolved gases, and allowed to solidify to test for possible re-crystallization. The samples were analyzed before and after the measurements (solid before melting and solid after cooling) by scanning electron microscopy–energy-dispersive X-ray spectroscopy (SEM–EDS) using a Hitachi S2400 electron microscope with X-ray detector Rontec Standard (Microscopy Laboratory, DEM, Instituto Superior Técnico), and compositions did not show appreciable differences from the compositions displayed in Table 1. Quality checks of all alloys were performed by DSC, using a TG-DSC111 from Setaram, France, determining the melting/freezing zones (in heating and cooling). The DSC was calibrated in enthalpy and temperature, according to procedures described previously [16,17].

Figure 4 shows one of such thermograms, obtained for alloy VII, proving that there are no impurities melting outside the expected zone, and that no solid-state phase transitions were present, except for a small perturbation at 262 °C that can be identified in the phase diagram (Fig. 3) as the appearance of a ZnAl compound dissolved in molten Zn. The freezing zone for this alloy is 379.30 °C to 372.51 °C. All other alloys showed the same behavior. Table 1 also displays the zones, detected as the onset temperatures for the first solidification detected, T_{liq} (*liquidus* line) and the complete freezing, T_{sol} (*solidus* line).

An estimate of the density of each alloy at each of the measured temperatures is necessary for the viscosity determination. Again, in the absence of experimental data, the densities of the alloys were estimated, using the composition furnished. If the alloys can be considered a “quasi” binary system, then the atomic volumes for the alloy can be estimated from the atomic volumes of the pure components by the following expression:

$$V = \sum x_i V_i \tag{7}$$

I J K

Fig. 2 General view of the viscometer equipment, including the inert atmosphere and vacuum systems: (A) oscillating initiator connected to the Pt92/W8 suspension wire; (B) He–Ne laser and photodetectors, on a vibration-free table; (C) high-temperature furnace; (D) temperature measurement thermocouple; (E) furnace temperature controller; (F) inert gas intake; (G) vacuum system; (H) vibration-free table; (I) inertial disk and mirror; (J) quartz window for laser beam; and (K) layout of the electronic instruments: timer interval counter, multimeter, and computer for data acquisition and control

Table 1 Composition of the Zn–Al alloys, in mass%

Alloy	Al (mass%)	Mg (mass%)	Mn (mass%)	Pb (ppm)	Si (ppm)	Fe (ppm)	Freezing interval $T_{\text{liq}} - T_{\text{sol}}$ (°C)
I	5	<50 ppm	0	0	100	25	380.73 – 363.65
II	5	<50 ppm	0	500	100	25	385.76 – 350.20
III	5	0.02	0	500	100	25	380.00 – 374.81
IV	5	0.05	0	500	100	25	385.33 – 350.47
V	5	<50 ppm	0.05	500	100	25	386.34 – 369.23
VI	5	<50 ppm	0.1	500	100	25	386.07 – 371.70
VII	5	<50 ppm	0	1000	100	25	379.30 – 372.51
VIII	4.5	<50 ppm	0	500	100	25	384.93 – 373.38

Minor components are displayed in parts per million (ppm)

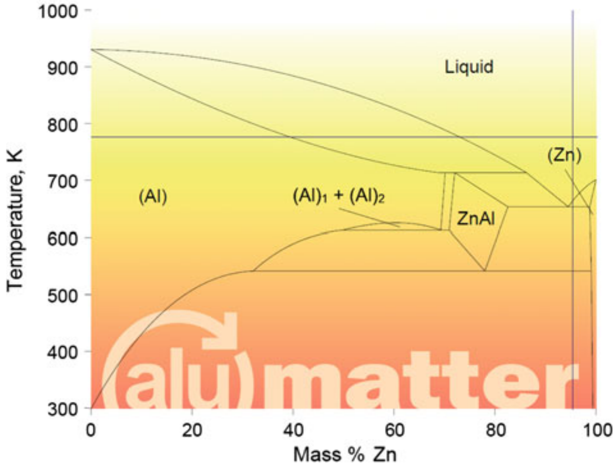


Fig. 3 Binary Zn–Al phase diagram, at 95 % Zn. Schematic diagram © 2001–2009 MATTER, The University of Liverpool (http://aluminium.matter.org.uk). *Vertical line* displays the 5 % Al composition, very close to the eutectic composition

which is equivalent for a multicomponent alloy to

$$\frac{1}{\rho} = \frac{x_{\text{Zn}}}{\rho_{\text{Zn}}} + \frac{x_{\text{Al}}}{\rho_{\text{Al}}} + \sum_{j=3}^{N} \frac{x_j}{\rho_j} \tag{8}$$

In this equation, ρ is the density of the alloy, ρ_{Zn} and ρ_{Al} are the densities of pure zinc and aluminum, respectively, at the same temperature and pressure of the alloy, and x_{Zn} and x_{Al} are the molar fractions of the main metals in the alloy. The summation for $j = 3$ to the total number of components in the alloy, N (a maximum of 7), accounts for the presence of the other minor components. As the uncertainty of the density prediction was estimated to be of the order of 1 %, elements with compositions

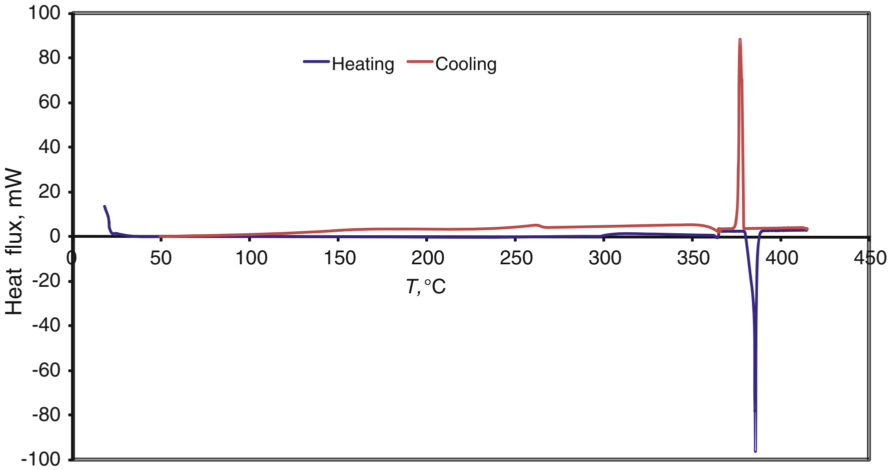


Fig. 4 DSC thermogram of alloy VII, on heating and cooling (scanning rate near melting or freezing $5\ ^\circ\mathrm{C}\cdot\mathrm{min}^{-1}$, stainless steel crucible, air). Freezing zone is 379.30 °C to 372.51 °C

Table 2 Thermal properties of elements used in the estimation of the density of the alloys using Eqs. 8 and 9 [19]

	T_m	$\Lambda\ (\mathrm{kg\cdot m^{-3}\cdot K^{-1}})$
Zn	692.7	0.98
Al	933.5	0.35
Mg	922	0.26
Si	1683	0.35

not greater than 0.5 % will not contribute significantly to the current estimation. This calculation is very reliable, as the density of this kind of binary alloy does not usually deviate from the simple weighted average of the densities of the constituent elements [18]. The uncertainty in density results in a significant contribution to the viscosity uncertainty budget [9,13,14].

The density of the pure components was calculated from their known dependence on temperature, as it was found experimentally that the temperature dependence of density for liquid metals and alloys is linear and can be represented by

$$\rho = \rho_\mathrm{m} + \Lambda(T - T_\mathrm{m}) \tag{9}$$

where ρ_m is the density of a pure liquid at the melting point and Λ is the temperature-dependence coefficient. Table 2 shows the necessary data for the calculation of the density of the pure metals (Zn, Al, and Mg) and Si, those which are important for the calculation of the density of the alloys under study [19].

3 Results and Discussion

The viscosity measurements were performed at four nominal temperatures, and the data are presented in Table 3. Each result presented is a mean of at least three

Table 3 Experimental measurements of the viscosity of different alloys

Alloy	T (K)	T (°C)	η (mPa · s)	Estimated uncertainty (%)
I	689.43	416.28	3.247	2–4
	719.24	446.09	2.860	
	750.08	476.93	2.725	
II	690.31	417.16	2.428	2–4
	707.76	434.61	2.265	
	719.15	446.00	2.119	
	749.98	476.83	1.875	
III	689.59	416.44	2.595	2–4
	720.67	447.52	2.433	
	751.61	478.46	2.316	
IV	689.60	416.45	2.655	2–4, except at the lowest temperature (5)
	720.24	447.09	2.446	
	751.80	478.65	2.314	
V	690.08	416.93	2.523	2–4
	751.29	478.14	2.244	
VI	690.40	417.25	2.425	3–5
	703.55	430.40	2.290	
	718.44	445.29	2.259	
	750.39	477.24	1.576	
VII	690.72	417.57	2.004	3–4
	703.36	430.21	1.849	
	719.88	446.73	1.810	
	751.50	478.35	1.731	
VIII	690.98	417.83	2.715	3–5
	702.88	429.73	2.619	
	749.44	476.29	2.087	

measurements. Figure 5 shows the measurements obtained for the studied alloys. The viscosity measurements of Zn–Al alloys are scarce, and we were able to find only one study on this kind of material in recent years [18]. In a recent review [20] on data and models for the viscosity of pure elements and alloys, several models to correlate the viscosities of binary or ternary alloys were discussed. Among these, the Chhabra model [21] seems to be appropriate to describe the variation of the viscosity of alloys with temperature. For a pure molten metal, the viscosity can be given by

$$\log(\eta + 1) = 10^{b_1} T^{b_2} \tag{10}$$

where b_1 and b_2 are characteristic constants for each element, η is the viscosity of the melt in mPa · s, and T is the temperature in K. Table 4 shows the values of b_1 and b_2 for pure Zn and Al [21]. This equation gives a better description of viscosities compared with the classical Arrhenius equation. For multicomponent mixtures the equation was

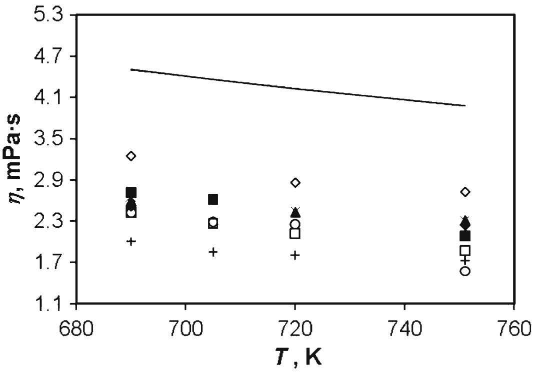


Fig. 5 Viscosity of the molten alloys as a function of temperature: ◇ alloy I; □ alloy II; ▲ alloy III; × alloy IV; ◆ alloy V; ○ alloy VI; + alloy VII; ■ alloy VIII. Line shows the calculated viscosity according to the Chhabra model [21]

Table 4 Parameters of the Chhabra equation [21]

Metal	Temperature range (K)	b_1	$-b_2$
Aluminum (Al)	935.15–1156.15	2.755	1.0740
Zinc (Zn)	693.15–1096.15	2.711	1.0151

modified [22], and the coefficients b_1 and b_2 for the alloys are obtained from the usual linear mixing rules, by the following expressions:

$$\begin{aligned} b_{1,\text{alloy}} &= \sum_{i=1}^{\text{N}} x_i b_{1,i} \\ b_{2,\text{alloy}} &= \sum_{i=1}^{\text{N}} x_i b_{2,i} \end{aligned} \tag{11}$$

As can be seen, if we consider the molten alloys as a quasi-binary alloy ($N = 2$), the calculated values from the Chhabra model will be greater than the measured values. This is also displayed in Fig. 5. If we introduce in Chhabra's equation the molar fractions of minor components, no important differences were found in calculated viscosities, because the mole fractions are very small. So the effect of minor components is difficult to model, and current equations are not able to explain the variation of viscosity with temperature for these complex multicomponent mixtures. These observations are not really unexpected, as we have to extrapolate data to temperatures lower than the melting points of some of the components, namely, aluminum.

In Figs. 6, 7, 8, and 9, the viscosities of similar alloys are displayed, when the composition of only one of the elements is changed, at rounded temperatures, 690 K, 705 K, 720 K, and 751 K. Figure 6 shows that the addition of Pb decreases the viscosity (alloys I, II, and VII). This could be an important effect in enhancing the castability of the alloys, for instance, optimizing mold-filling aspects and reducing the porosity. Figure 7 shows the effect of adding Mg, while keeping other constituents (namely Pb) constant (alloys II, III, and IV). In this case, the viscosity increases, almost

Fig. 6 Effect of lead (Pb) addition on the viscosity of molten alloys I, II, and VII. Marks are the same as in Fig. 5

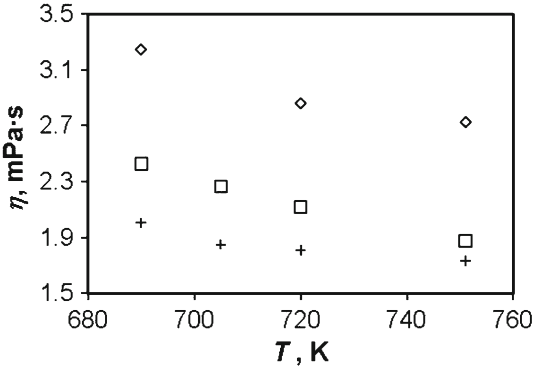


Fig. 7 Effect of magnesium (Mg) addition on the viscosity of molten alloys II, III, and IV. Marks are the same as in Fig. 5

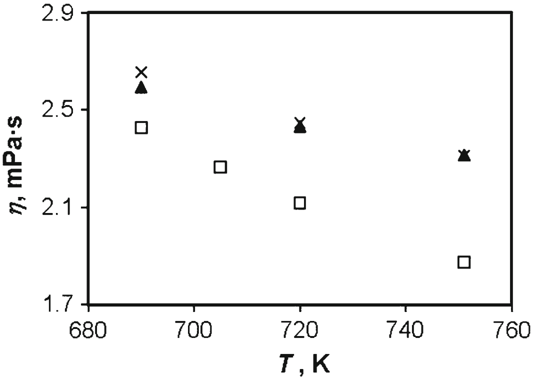
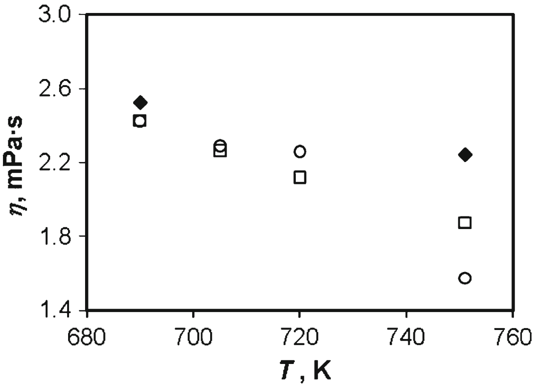


Fig. 8 Effect of manganese (Mn) addition on the viscosity of molten alloys II, V, and VI. Marks are the same as in Fig. 5



independently of the amount of Mg added. Figure 8 shows that small amounts of Mn do not have a significant effect on the viscosity of the molten alloys, except at the highest temperature. As expected, an increase of the Al content (from 4.5 % to 5 %) reduces the viscosity of the alloys, as can be seen in Fig. 9. Effects of this type were also found for aluminum-based alloys [23].

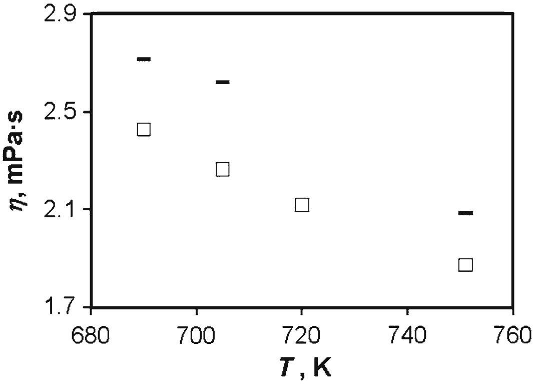


Fig. 9 Effect of aluminum content on the viscosity of molten alloys II and VIII. Marks are the same as in Fig. 5

As a final remark, we would like to say that all the alloys showed Newtonian behavior in the temperature range studied and the temperature dependence of the viscosity is non-Arrhenius, as usually found for pure molten metals. For most of the alloys and within the uncertainty of the experimental results, the viscosity varies linearly with temperature (II, III, IV, and VI). This is possibly caused by the complex variation of the multicomponent liquid viscosity with temperature, already found for Al alloys [21,23]. Further investigations on more extended ranges are necessary to ascertain the validity of this behavior.

4 Conclusions

Viscosity measurements of Zn–Al alloys were performed with an oscillating-cup viscometer, in the molten state, as a function of temperature. The lowest temperature was 416 °C and the highest was 641 °C. The uncertainty of the measurements varies between 2 % and 5 %. Zn and Al are very important materials in die casting and galvanizing processes, as Al is important in increasing the fluidity, and also offers an important protection against steel corrosion. We concluded that Pb induces a decrease on the viscosity, with Mg having the opposite effect, while the effect of Mn is not significant.

All the alloys showed Newtonian behavior in the temperature range studied and that the temperature dependence of viscosity is non-Arrhenius, as usually found for pure molten metals.

As a result of this study, a completely new field of research was initiated in this laboratory. We hope to contribute to further knowledge and advances in this area, and we anticipate reporting new data on the viscosity of other high-temperature Zn–Al alloys in the near future.

Acknowledgments The alloys used in this study were produced by Umicore, which contracted "Centro de Ciências Moleculares e Materiais - FCUL" in 2004 to measure the alloy's viscosity at given temperatures. This publication was possible after the confidentiality period expired. We thank Arcelor Research Industry, Gent, Belgium, and namely, Dr. Serge Classens, for the permission granted to publish these data.

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