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EXPERIMENTAL INVESTIGATION AND CALCULATION OF SHEAR RATE, SHEAR STRESS AND POWER FOR MIXING OF SEMI-SOLID MIXTURES OF ZA27 ALLOY AND ZA27/Al_2O_3 COMPOSITES WITH LARGE Al_2O_3 PARTICLES

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Abstract

Shear rate, shear stress and power for mixing of semi-solid mixtures (SSMs) of ZA27 alloy and ZA27 alloy-based composites with large Al_2O_3 particles were investigated. Mixing was carried out using cylindrical and paddle stirrer. Mixing power was determined by electric method in the temperature range from 479 to 440 ˚C at constant cooling rate. Calculated values of shear rate, shear stress and mixing power for the composite (SSMs) are lower than those of ZA27 alloy in the whole range of operating temperatures.

Key words: ZA27 alloy; metal-matrix composites; semi-solid mixtures; shear rate; shear stress; mixing power

Introduction

It has been known that mechanical characteristics of zinc-based ZA27 alloy (with 25 wt. % Al and 3 wt.% Cu) deteriorate at higher operating temperatures (> 80 ˚C) [1, 2]. However, it was shown that particulate composites with base ZA27 alloy preserved favorable mechanical characteristics at higher temperatures [1]. Recently, It has been reported that composites with base ZA27 alloy and large Al_2O_3 particles (250 μm), produced by compocasting process, are distinguished by good combination of mechanical and tribological properties [3, 4]. A favorable distribution of reinforcing

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particles in the matrix alloy can be achieved by additional mixing of semi-solid mixture (SSM) after particles infiltration in the compocasting process. Semi-solid processing of an alloy has been usually accompanied by the related rheological investigations. Rheological investigations of the composites SSMs and determination of mixing power as a function of SSMs viscosity enable improvement of the composites processing as well as a proper design of processing equipment. Rheological characteristics of SSMs have been usually examined by using viscosimeters. SSMs were exposed to shear stress in the extremely narrow gaps between the static and rotating parts of a viscosimeter [5−7]. Mixing was carried out at a nearly constant value of shear stress while controlling values of shear rate. Thus, it was possible to calculate rheological parameters of SSMs with great accuracy. In addition, another rather different approach to the investigations of rheological behavior of SSMs was also applied [8]. Namely, the rheological characteristics of a hypereutectic Al–Si alloy were investigated in the induction vacuum furnace [8], where a distance from the stirrer to the wall of crucible was significantly larger than in the viscosimeters with gaps.

Lehuy et al. examined rheological behavior of ZA27 alloy SSMs [5], but there have been no results reported so far concerning rheological characteristics of ZA27 alloy-based composites. This is in contrast to numerous published papers referring the rheological studies of aluminum alloys, as well as aluminum alloys based composites [6−8].

Changes of apparent viscosity, which is the basic rheological characteristic of ZA27 alloy SSM and SSMs of ZA27 alloy-based composites, were determined in our previous work [9] in the mixing conditions close to real ones, using the so-called "method of measuring input power for commutator motor" [10] (electric method in the further text).

The investigations have been continued within this work and calculation of important mixing parameters of SSMs of ZA27 alloy and ZA27 alloy based composites such as shear rate, shear stress and mixing power, has been evaluated. SSMs mixing was carried out using the electric method. An attempt was also made to establish the correlation between the values of mixing power obtained by the electric method and applying theoretical expression [11].

Experimental

Thixocast samples of ZA27 alloy were used in rheological experiments as well as samples of ZA27 alloy-based composites with large Al_2O_3 particles. The composites were produced by compocasting technique with addition of 3, 8 and 16 wt. % Al_2O_3 (250 μm particles size) into the SSM of the matrix alloy. Mixing experiments were performed using the apparatus shown in [9].

The alloy was melted and preheated up to 550 °C to clean the slag from the melt surface. The melt was left to cool down to 485 °C at 5 °C min^{-1} (approximately isothermal regime) when the active part of the stirrer was immersed into the SSM of ZA27 alloy as well as into the SSMs of the composites. Mixing of ZA27 alloy SSM was carried out using both cylindrical and paddle stirrer, while SSMs of the composites were mixed using paddle stirrer only. The cylinder (d = 4 cm; h = 10 cm) was made of Al_2O_3, while the steel paddle (4×0.2×10 cm) was coated with an Al_2O_3 layer. Mixing was performed at 450 rpm. The active part of stirrer was immersed into the SSM of ZA27 alloy as well as into the SSMs of the composites at 485 °C. The increase in stirrer rotation frequency was gradual until the selected frequency was reached. The selected rotation frequency 450 rpm was achieved when the SSM was cooled at 479 °C, what was the starting temperature of the controlled mixing process. In the temperature range from 479 to 440 °C the change of electric current intensity during mixing at spontaneous cooling (5 °C min^{-1}) was measured at every 3 °C of the temperature decrease. Each value of electric current intensity is given as an average of 7 to 10 measured values.

The electric method was used for monitoring changes of current intensity during stirrer rotation in the air at 450 rpm, as well as for monitoring changes of current intensity during SSMs mixing (of ZA27 alloy and composites) at slow cooling rate. In fact, changes of total electric current intensity during mixing at stabilized and constant network voltage were measured. The difference between the value of current intensity for SSM mixing at any operating temperature (479 to 440 ºC) and the value of current intensity during stirrer rotation in the air is the effective current intensity necessary to overcome the medium resistance to flow. This is effective mixing current. Values of mixing power were determined on the basis of experimental results obtained by the electric method (Appendix 2). Values of basic rheological parameters of SSMs were calculated afterwards.

Results and discussion

Solid fraction

Applying cooling rate of 5 ˚C min^{-1} for the ZA27 alloy SSM the cooling regime can be considered as nearly "isothermal". Changes of ZA27 alloy solid fraction with temperature during SSM solidification were determined on the basis of aluminum–zinc equilibrium phase diagram shown in Figure 1 [12] using the lever rule.

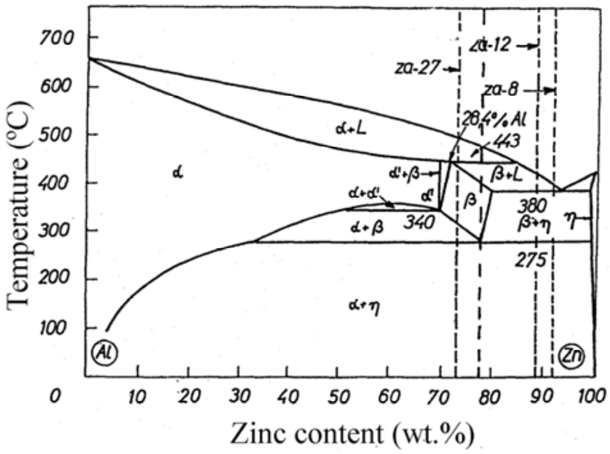

Figure 1. Aluminum–zinc equilibrium phase diagram [11]

Values of solid fraction in the SSM of matrix alloy and SSMs of composites depending on temperature were obtained on the basis of experimental data and are shown in Figure 2.

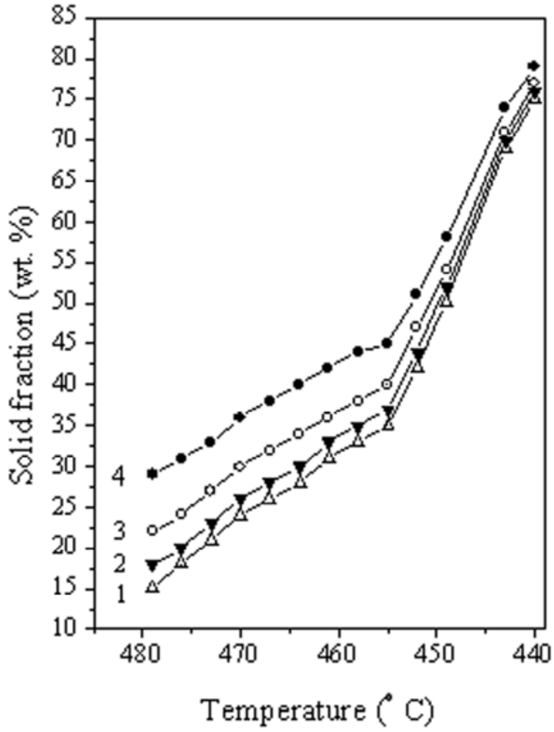

Figure 2. Solid fraction dependence on temperature during isothermal solidification. 1) SSM of ZA27 alloy; 2, 3, 4) SSMs of composites with 3, 8 and 16 wt. % Al_2O_3, (250 µm particle size), respectively.

Solid fraction depending on temperature during "isothermal" cooling of the ZA27 alloy SSM is presented by curve 1 in Figure 2. These results are in accordance with Lehyu's results [5] for SSM solidification of the same alloy at similar value of cooling rate.

Total solid fraction of the composites SSMs in the two-phase semi-solid region is changed as a function of the amount of infiltrated reinforcing particles. Large Al_2O_3 particles used as strengthening particles within this work are chemically and thermodynamically inert in the SSM of ZA27 alloy. At any temperature in the semi-solid temperature range the total solid fraction of a composite SSM consists of primary particles of α phase, originating from the matrix ZA27 alloy and reinforcing Al_2O_3 particles.

Mixing parameters

Shear rate and shear stress

Shear rate γ can be expressed as a ratio between stirrer frequency n and geometric parameter k according to Equation 1. The derivation of Equation 1 is given in Appendix 1.

$$\gamma = \frac{4 \cdot \pi \cdot n}{1 - k^2} \quad (1)$$

Where: k is the ratio between stirrer diameter and crucible diameter ($k = d/D$). For the cylindrical stirrer diameter d = 4 cm and crucible diameter D = 7.5 cm (used within this work) the value of k was 0.53. The same value of k was obtained in the case of paddle stirrer, for diameter (d = 4 cm) of paddle stirrer traced by the active part of stirrer during rotation (d = 4 cm), according to Greif [11]. Stirrer frequency rotation n was kept constant in all experiments (450 rpm) and calculated value of shear rate (Equation 1) was 131 s^{-1} so that results obtained within this work can be related with results of other authors who used Brookfield [5] and Searle viscosimeter [7], or applied a torquemeter [8].

Values of shear stress τ (Pa) during mixing of SSMs (ZA27 alloy and composites) were calculated as a dependence on total solid fraction according to Equation 2:

$$\tau = \eta \cdot \gamma \quad (2)$$

Values of η and γ are given in [9]. The derivation of Equation 2 is also presented in Appendix 1.

Calculated values of shear stress for the SSM of ZA27 alloy and SSMs of the composites are presented in Table 1.

Table 1 Calculated values of shear stress as dependence of total solid fraction of SSM

Total solid fraction (wt. %)	Shear stress (Pa)				
	ZA27 alloy		Composites (paddle stirrer)		
	cylindrical stirrer	paddle stirrer	ZA27+3wt. % Al_2O_3	ZA27+8wt. % Al_2O_3	ZA27+16 wt.% Al_2O_3
24	191	652	451	479	/
26	310	720	484	493	/
28	371	772	529	508	409
31	503	862	605	538	445
33	623	1011	638	558	463
35	850	1186	706	608	463
42	1006	1350	997	867	694
50	1089	1466	1140	1067	1104
69	1724	2328	1388	1256	1662

Values of shear stress in Table 1 were obtained by multiplying apparent viscosity values [9] with the value of shear rate that was kept constant within all experiments (γ =131 s^{-1}). It can be seen that values of shear stress increase with growth of total solid fraction. Values of shear stress when using paddle stirrer are higher comparing to those of cylindrical stirrer. In the presence of infiltrated Al_2O_3 particles (composites SSMs) values of shear stress are lower than for ZA27 alloy SSM and decrease with increase of reinforcing particles fraction.

Calculated values of shear stress presented in Table 1 are in accordance (same order value) with the results reported by Spencer and al. [13] for SSM of Sn-15Pb alloy.

Flow regime during SSMs mixing

The basic criterion for evaluation of flow regime during SSMs mixing within this work was the value of Reynolds number. For mixing in a cylindrical vessel when using a central rotating paddle, turbine or propeller, the characteristic dimension is diameter of agitator *d*. Then according to [11, 14] the Reynolds number *Re* may be expressed as:

$$Re = \frac{n \cdot d^2 \cdot \rho}{\eta} \tag{3}$$

where: *d* - equivalent diameter of stirrer, i.e. diameter of circle traced by an active part of stirrer (m), ρ - density of medium (kg m^{-3}), η - apparent viscosity of medium (Pa s).

Stirrer rotation frequency *n* and equivalent diameter of stirrer *d* were identical in all experiments. Density of medium is 5000 kg m^{-3} in the case of ZA27 alloy, while density of Al_2O_3 particles is 3910 kg m^{-3}. Concerning composites, SSMs density of medium is calculated according to the following expression:

$$\rho_{CM} = \rho_{ZA27} \cdot \upsilon_{ZA27} + \rho_{Al_2O_3} \cdot \upsilon_{Al_2O_3} \tag{4}$$

where: ρ_{CM}- density of composite (kg m^{-3}), ρ_{ZA27}- density of ZA27 alloy (kg m^{-3}), ρ_{Al2O3}- density of Al_2O_3 particles (kg m^{-3}), υ_{ZA27}- volume fraction of ZA27 alloy, υ_{Al2O3}- volume fraction of Al_2O_3 particles.

Table 2 Calculated values of Reynolds number as dependence of total solid fraction of SSM

Total solid fraction (wt. %)	Reynolds number Re			
	ZA27 alloy (paddle stirrer)	Composites (paddle stirrer)		
		ZA27 + 3 wt. % Al_2O_3	ZA27 + 8 wt. % Al_2O_3	ZA27 + 16 wt. % Al_2O_3
24	12.1	17.4	16.1	/
26	10.9	16.2	15.6	/
28	10.3	14.8	14.9	18.5
31	9.2	12.7	14.5	17.1
33	7.7	12.2	13.8	16.3
35	6.6	11.1	12.7	16.3
42	5.8	7.8	8.9	10.9
50	5.4	6.8	7.3	7.1
69	3.5	5.7	6	4.5

Reynolds number for SSMs is designated as $Re^{\bullet}$ in the further text considering that examined SSMs are non-Newtonian fluids. $Re^{\bullet}$ values during mixing of the SSMs were calculated using Equation 3, as a function of total solid fraction and presented in Table 2. Total solid fraction increases during SSMs mixing at slow cooling as well as the values of apparent viscosity causing a decrease of Reynolds number.

On the basis of calculated $Re^{\bullet}$ values (Table 2) one can conclude that mixing of SSMs in all cases was performed in the laminar flow regime. $Re^{\bullet}$ values were used for calculation of mixing power.

Mixing power

Current intensity and mixing power during SSMs mixing using paddle stirrer are related by the following well-known expression:

$$N = U \cdot \Delta I \cdot \cos\varphi \quad (5)$$

where N denotes mixing power (W), while other terms are as in Appendix 1. Values of network voltage U and power factor of stirrer electromotor $\cos\varphi$ are constant and consequently mixing power is dependent on the current intensity change:

$$N = f(\Delta I) \quad (6)$$

Changes of electric current intensity ΔI during mixing of SSMs (ZA27 alloy and composites) were measured as it was described in section 2 and the results are presented in Tables A.2.1 and A.2.2 in Appendix 2. Current intensity changes are dependent on total solid fraction of SSMs, as can be seen in Tables A.2.1 and Table A.2.2. Values of mixing power for SSMs mixing (paddle stirrer) were calculated on the basis of experimentally obtained ΔI values using Equation 5. These values of mixing power are presented in Figure 3 (a – d) (curve 2).

Input mixing power can be calculated using theoretical expression [14]:

$$N = C \cdot \rho \cdot n^3 \cdot d^5 \quad (7)$$

where C denotes a power factor. This term is dependent on the fluid flow regime and stirrer type while other terms in Equation 7 are the same as in the previous section (see *Flow regime during SSMs mixing*). The values of SSM density ρ, stirrer rotation frequency n and equivalent diameter of stirrer d were identical within all experiments. A nomograph for laminar fluid flow and paddle stirrer [11] was used to determine values of power factor C. These values were determined for each $Re^{\bullet}$ value, on the basis of calculated $Re^{\bullet}$ values (Table 2 in section 3.2.2). Values of power factor C were inserted in Equation 7 and appropriate values of mixing power were calculated for SSMs (ZA27 alloy and composites). These values are shown in Figure 3 a - d (curve 1) as dependence on total solid fraction. $Re^{\bullet}$ values lower than 5 were not given in nomograph [11] and consequently the corresponding values of mixing power are not shown in Figure 3. It can be observed that results obtained on the basis of theoretical expression (Equation 7 [14]) and by using the electric method are in good agreement, particularly when total solid fraction is lower than 50 wt. %.

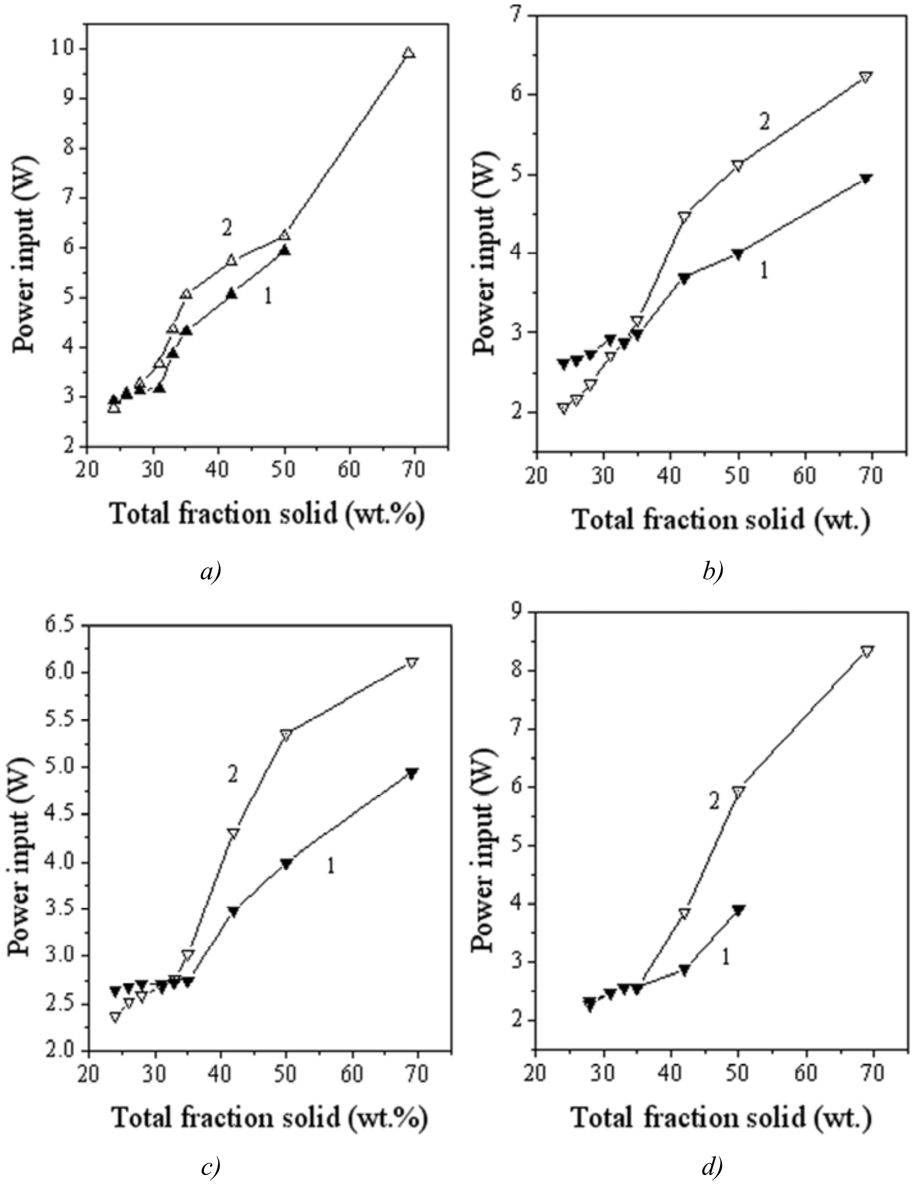

Figure 3. Mixing power dependence on total solid fraction during SSMs mixing: 1) According to the theoretical expression [14]; 2) Electric method. a) ZA27 alloy, b) ZA27 + 3 wt. % Al_2O_3, c) ZA27 + 8 wt. % Al_2O_3, d) ZA27 + 16 wt. % Al_2O_3.

In general, the results reported in this paper explicitly showed that used electric method proved to be a reliable, reproducible and rather accurate method in determining values of basic rheological parameters and mixing power of SSMs.

Conclusions

1. Infiltration of large Al_2O_3 particles (250 μm particle size) into the SSM of ZA27 alloy causes decrease of SSM shear stress. Values of shear stresses for composites SSMs decrease as the fraction of reinforcing Al_2O_3 particles increases. This is not valid for the composite SSM containing 16 wt. % Al_2O_3 particles when total solid fraction exceeds 50 wt. %.
2. During SSMs mixing values of shear stress and mixing power were higher when using paddle stirrer than those when cylindrical stirrer was used.
3. Values of mixing power increase with growth of total solid fraction, i.e. the mixing power increases with decrease in temperature. Values of mixing power at constant temperature are reduced with increase in mass fraction of reinforcing particles.
4. Mixing of SSMs of ZA27 alloy and composites was performed in the laminar flow regime as was indicated by the calculated values of Reynolds number. $Re^{\bullet}$ values decrease as the total solid fraction increases, that is with decrease in temperature $Re^{\bullet}$ values also decrease. At constant temperature $Re^{\bullet}$ values become higher with an increase in fraction of reinforcing particles
5. The used electric method enabled acquisition of experimental data for approximate calculation of the most important mixing parameters of the SSMs of ZA27 alloy-based composites with large Al_2O_3 particles. In general, this method could be very useful for evaluation of rheological behavior of composites SSMs.

Appendix 1 *Expressions for calculation of basic rheological parameters*

a) Mixing power and rotation momentum

$$N = U \cdot \Delta I \cdot \cos\varphi \tag{1.1}$$

$$M = N/\omega \tag{1.2}$$

$$\omega = 2 \cdot \pi \cdot n \tag{1.3}$$

Where:
N - power of mixing (W)
U - network voltage (V)
ΔI - mixing current intensity (A)
$\cos\varphi$ - power parameter
M - rotation momentum (momentum of mixing) (N m)
ω - angular velocity of stirrer (rad s^{-1})
n - stirrer frequency (s^{-1})

b) Shear rate and shear stress

The well-known expression for rotation momentum is as follows:

$$M = F \cdot r \tag{1.4}$$

Where:
F - shear force (N),
r - radius of circle traced by the active part of stirrer (m),

Inserting (1.4) in the equation for apparent viscosity [9] the following expression is obtained:

$$\eta = \frac{F \cdot r \cdot (1-k)^2}{4 \cdot \pi \cdot r^2 \cdot L \cdot \omega} \tag{1.5}$$

Considering that contact area A (m^2) between the active part of stirrer and SSM is as follows:

$$A = 2 \cdot r \cdot \pi \cdot L \tag{1.6}$$

The expression (1.5) can be written as:

$$\eta = \frac{F \cdot (1-k^2)}{2 \cdot A \cdot \omega} \tag{1.7}$$

Having in mind the expression (1.3) and that shear stress $\tau = F/A$ it turns out:

$$\eta = \tau \cdot \frac{(1-k^2)}{4 \cdot \pi \cdot n} \tag{1.8}$$

Accordingly, the expression for shear stress is as follows:

$$\tau = \eta \cdot \frac{4 \cdot \pi \cdot n}{1 - k^2} \tag{1.9}$$

In laminar flow regime shear stress can be expressed as:

$$\tau = \eta \cdot \gamma \tag{1.10}$$

where γ denotes shear rate (s^{-1}).
Inserting (1.9) in (1.10) the expression for shear rate is obtained:

$$\gamma = \frac{4 \cdot \pi \cdot n}{1 - k^2} \tag{1.11}$$

Appendix 2 *Changes of current intensity during mixing*

Table A.2.1 Changes of electric current intensity as dependence of temperature during SSM mixing of ZA27 alloy

t (°C)	Solid fraction (wt. %)	I_c (A)	ΔI_c (A)	I_p (A)	ΔI_p (A)
440	75	0.516	0.236	0.478	0.176
443	69	0.424	0.144	0.402	0.100
449	50	0.376	0.096	0.365	0.063
452	42	0.364	0.084	0.360	0.058
455	35	0.351	0.071	0.353	0.051
458	33	0.332	0.052	0.346	0.044
461	31	0.322	0.042	0.339	0.037
464	28	0.311	0.031	0.335	0.033
467	26	0.306	0.026	0333	0.031
470	24	0.296	0.016	0.330	0.028
473	21	0.295	0.015	0.329	0.027
476	18	0.295	0.015	0.330	0.028
479	15	0.294	0.014	0.328	0.026

I_c- total current intensity during mixing with cylindrical stirrer (A),
I_p- total current intensity during mixing with paddle stirrer (A),
I_{ca} = 0.280A- current intensity measured when cylindrical stirrer rotates in the air,
I_{pa} = 0.302A - current intensity measured when paddle stirrer rotates in the air,
ΔI_c, ΔI_p - effective current intensity (A), i.e. is the difference between current intensity drawn by the stirrer (cylinder or paddle) electromotor from electric network during SSM mixing, and current intensity drawn from electric network when the stirrer rotates in the air:

$\Delta I_c = I_c - I_{ca}$

$\Delta I_p = I_p - I_{pa}$

By analogy with previously stated, values of current intensity changes during mixing of the composites SSMs were calculated and presented in Table A.2.2.

Table A.2.2 Changes of current intensity as dependence of total solid fraction during SSMs mixing of ZA27 alloy and composites

Total solid fraction (wt. %)	ΔI_p (A)			
	ZA27	ZA27+3 wt.% Al_2O_3	ZA27+ 8 wt.% Al_2O_3	ZA27+16 wt.% Al_2O_3
24	0.028	0.021	0.024	/
26	0.031	0.022	0.025	/
28	0.033	0.024	0.026	0.023
31	0.037	0.027	0.027	0.025
33	0.044	0.029	0.028	0.026
35	0.051	0.032	0.030	0.026
42	0.058	0.045	0.043	0.039
50	0.063	0.049	0.053	0.062
69	0.100	0.063	0.063	0.093

ΔI_p - effective current intensity (A) i.e. is the difference between current intensity drawn by the stirrer (paddle) electromotor from electric network during SSM mixing, and current intensity drawn from electric network when paddle stirrer rotates in the air.

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