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RHEOLOGICAL ANALYSIS OF SEMI-SOLID A380.0 ALUMINIUM ALLOY

ANALIZA WŁAŚCIWOŚCI REOLOGICZNYCH STOPU ALUMINIUM A380.0 W STANIE STAŁO-CIEKŁYM

Knowledge of the rheological properties is crucial for the numerical modeling of technological processes. The main objective of this study was to conduct an analysis of the rheological properties of A380.0 (AlSi9Cu3(Fe)) aluminium alloy in the semi-solid state. The results could be used for identification of temperature range of the alloy, where thixoforming processes could be executed. Another purpose of the experimental work could be development of the mathematical models of the alloy apparent viscosity. The significant achievement of this particular study is an application of a viscometer which was specially designed for material tests executed at high temperatures, such as the measurement of liquid or semi-liquid aluminium viscosity. This paper presents the results of a rheological analysis of aluminium alloy.

Keywords: thixoforming, rheological properties, viscosity, thixotropy, aluminium alloys

Wiedza dotycząca właściwości reologicznych jest istotna w zagadnieniach modelowania procesów technologicznych. Nadrzędnym celem pracy jest analiza właściwości reologicznych stopu aluminium A380.0 (AlSi9Cu3(Fe)) w stanie stało-ciekłym. Otrzymane wyniki mogą służyć do wyznaczenia zakresu temperatur, w którym analizowany stop może być poddawany procesowi formowania tiksotropowego. Dodatkowo wyniki te mogą być wykorzystane do opracowania matematycznego modelu lepkości pozornej badanego stopu. Jednym z osiągnięć tej pracy jest praktyczne zastosowanie lepkościomierza rotacyjnego do pomiaru lepkości ciekłego oraz stało-ciekłego stopu aluminium. W pracy przedstawiono wyniki pomiarów lepkości.

1. Introduction

Thixoforming is an innovative method of processing of metal alloys in the semi-solid state. Such shaping method has many advantages in comparison with classical metal forming and foundry processes. Thixoforming technologies are based on modified devices normally used for forging or casting processes. Nowadays, research is conducted with the practical application of this method in aluminium, magnesium and steel processing as its goal [1-7].

Numerical modelling of thixoforming processes requires knowledge of viscosity of cast metal alloys. It helps to determine the velocity field of the semi-solid metal inside the die. Precise determination of this field allows to predict the temperature distribution and avoid possible defects. The main goal of this paper is an analysis of the semi-liquid A380.0 (AlSi9Cu3(Fe)) alloy viscosity. The basic difficulty was very high temperature of semi-liquid alloy and it required the application of special equipment.

Moreover the research conducted allowed the rheological properties, which are necessary for the identification of the main technological parameter values of the metalforming processes, to be determined [4]. The phase transformation, connected with the solidification, results in the high sensitivity

of material properties to changes of the technological parameters.

This paper presents the results of a rheological analysis of A380.0 aluminium alloy. The chemical composition of this alloy is shown in Table 1. Chemical analysis was carried out using handheld XRF analyser.

TABLE 1
Chemical composition of A380.0 alloy in weight-%

Element	Si	Cr	Mn	Fe	Ni	Cu	Zn
ASTM-standard specification [8]	7,5-9,5	<0,5	<0,5	<1,3	<0,5	3,0-4,0	<3,0
Investigated alloy	8,61	0,08	0,3	<0,85	0,04	3,36	1,09

The first stage of investigation was devoted to identification of content of the solid phase in the material as a function of temperature. For this purpose the thermodynamic calculations was applied using special module of ProCAST software. The calculations required a knowledge of the chemical composition. The content of the solid phase as a function of temperature is shown in Fig. 1. The solid fraction calculation was achieved through level rule method. In order

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to do this, the software determined the phase diagram using the Gibbs principle of minimum free energy. The diagram (see Fig. 1) shows solidus and liquidus temperatures approximately 505°C and 585°C. The collapse of the solid fraction curve results from melting process of eutectic. The authors suspect that the measurement of the chemical composition and, what is worse, phase composition calculations are flawed. Chemical analysis was carried out at lateral surface of the billet, where composition can differ from rest volume of the sample. It can cause wrong thermodynamic calculations.

This paper is the second publication from thematically related series devoted to process for the conferment of a degree of *doktor habilitowany* on Dr. Krzysztof Sołek, pursuant to the rules laid down in the Act of 14 March 2003 on Academic Degrees and Title and Degrees and Title in the Arts with later changes (Republic of Poland law). This series concerns thixoforming of metal alloys.

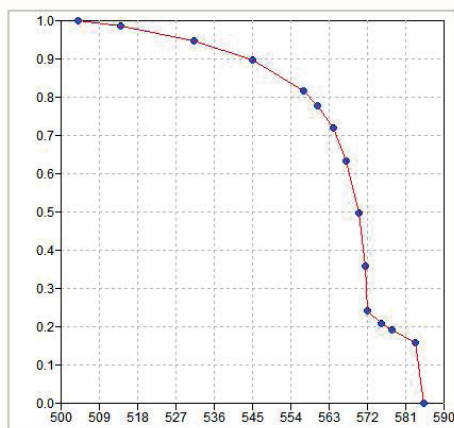


Fig. 1. Content of the solid phase in the A380.0 alloy as a function of temperature [°C]

2. Experimental set-up

The high temperature viscometer FRS1600 which, used for the rheological analysis, was designed by the Anton Paar company as a result of demand of the Faculty of Metals Engineering and Industrial Computer Science at the AGH University of Science and Technology in Krakow. Basically it consists of the head and the furnace which allows to obtain the temperature in the range 400-1500°C. Rheological analysis of aluminium alloys in the semi-solid state requires the use of a furnace where the work temperature is within the range of about 500 - 700°C.

The viscosity measurement of analysed alloy was performed using Searle's method [9-11]. It means that, this measurement was carried out using a rotational viscometer with a stationary cup (outer cylinder). In this method, the rod is set in motion and the cup is stationary (Figure 2). The cylinders are concentric, which means that both cylinders have the same symmetry axis (the rotation axis of the inner cylinder). In industrial laboratories, almost all rheometers work on this principle, which was named after G.F.C. Searle in 1912. The main disadvantage of this method is that turbulent flow conditions may occur while measuring of low-viscosity liquids at high rotational speeds. A photograph of the viscometer is shown in Figure 3.

Measurement of viscosity using FRS 1600 viscometer is limited to momentum of about 200 mNm. For security, in the case of measurement executed in this work this limit was reduced to 150 mNm. Viscosity measurement was conducted with the help of implemented procedure in the software using for control of the viscometer. At first, the samples were heated above liquidus temperature. Next the rotating rod was moved down inside crucibles with samples. After it the measurement procedure was started. During the measurement the protective atmosphere is blown into the furnace to avoid oxidation of the sample. The temperature of the sample could be precisely measured using thermocouple located directly below the crucible. After the completion of the measurement the sample is heated again above the liquidus temperature to eject the spindle from the crucible.



Fig. 2. Photograph of measurement tools (material - alumina) used in investigations



Fig. 3. Photograph of rotational viscometer used in investigations

3. Measurement of viscosity

The first step of the measurement concerned determination of relationship between alloy viscosity and the temperature. The basic rule indicates, that temperature decrease causes increase of registered viscosity, especially in the case of semi-solid state of metal alloy. This experiment allowed to identify the temperature range proper for thixoforming process. Analysed alloy can be subjected to thixoforging processes in the temperature range between 520 and 550°C. And the temperature of thixocasting processes range from 550 to 570°C. In the case of thixocasting the results of measurement can indicate the minimal temperature of material which allows for correct execution of the process. The criterion is the value of alloy viscosity, above which the material loses fluidity and complete filling of the die is not possible.

Next, for selected temperatures (530, 550, 560, 570°C) the measurements of viscosity versus shear rate were carried out. Changes of viscosity values were analysed in the range of the shear rate between 0.1 and 2,0s⁻¹. It should be mentioned that both the temperature changes and the shear rate changes were realized automatically according to the programmed procedure. Figure 4 shows the changes of viscosity of analysed aluminium alloy versus shear rate for different temperatures in which the semi-liquid state appears. Before each viscosity measurement, during change of the temperature, the sample were sheared with rate of about 5s⁻¹. An experience shows that viscometer is accurate within +/- 1.0% of the measurement range in use and have a repeatability with +/-0.2%. But, in real, the value of viscosity strongly depends on the temperature and morphology of the solid phase (state of microstructure).

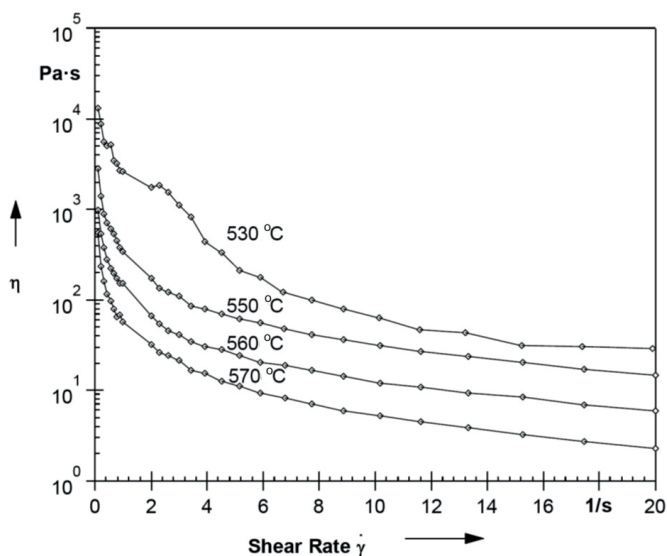


Fig. 4. Viscosity of analysed aluminium alloy versus shear rate for different temperatures

The shape of curves, describing relationship of the viscosity versus the shear rate, indicates the shear thinning behavior of the semi-solid aluminium slurry. It means the viscosity decreases with an increasing shear rate.

4. Identification of thixotropic properties

The second part of the experimental work was devoted to analysis of thixotropic properties. Thixotropy means time-dependent fluid behaviour in which the apparent viscosity decreases with time of shearing [12]. The viscosity of fluid usually recovers to initially value when shearing is stopped. The recovery process may take place over a long time. The rotational viscometer can be used for identification of thixotropy behaviour due to possibility of shear of the sample with constant rate during very long time. Figure 5 shows the changes of viscosity of analysed aluminium alloy versus time for different values of the shear rate. The basic rule indicates, that after increase of the shear rate, the viscosity decreases over a time, what can be observed in Figure 5. When the shear rate decreases one can observe an increase of the viscosity over a time. Before analysis of thixotropy properties, the semi-solid samples were sheared for some time to obtain globular microstructures with rate of about 5 s⁻¹.

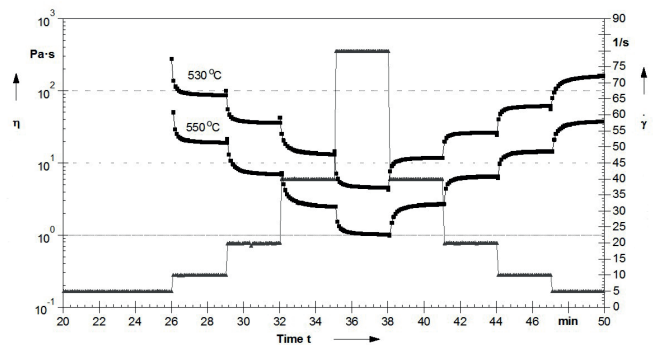


Fig. 5. Viscosity of analysed A380.0 aluminium alloy versus time for different values of shear rate and temperature

In the next step of experimental work the influence of the resting time on the yield stress was analysed (Figures 6, 8). At first, the samples were sheared for some time to obtain globular microstructures. Next, they were deformed in the several cycles. In each cycle, the sample was subjected to shear stress which increase with differ rate. Generally, lower rate of the shear stress increase gives longer resting time and higher yield stress (Figures 7, 9). These results mean that longer resting times cause microstructure transformation allowing to rebuild the solid skeleton. Such procedure was used in RWTH Aachen to investigate the thixotropy in the semi-solid metal alloys [13]. In this work the values of the yield stress were measured for 575 (Figures 6, 7) and 570°C (Figures 8, 9). The dots in Figures 6 and 8 associated with shear rate correspond with the moment of beginning of material flow. Value of the shear stress recorded in this moment corresponds to the yield stress.

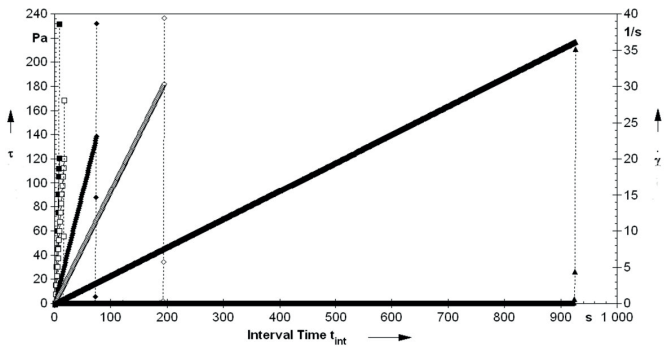


Fig. 6. Shear stress versus time for different delays before shearing for sample at 575°C

The measurements carried out in this work show an influence of the resting time on the yield stress in the semi-solid aluminium alloy.

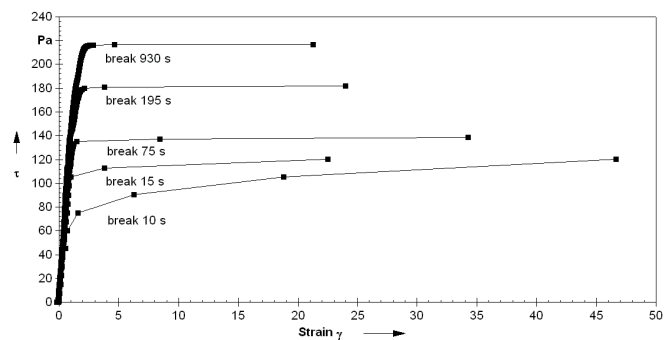


Fig. 7. Flow stress curves for different resting times at 575°C (“break” means resting time)

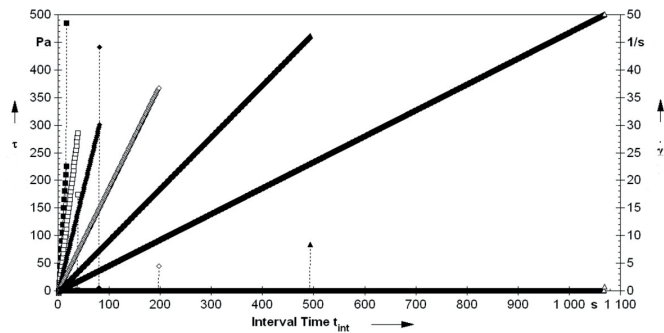


Fig. 8. Shear stress versus time for different delays before shearing for sample at 570°C

A comparison of the yield stress values for different values of the resting time was shown in Figure 10. The enclosed graph clearly show, that a decrease of the temperature causes an increase of the yield stress value. This effect results from greater amount of solid fraction in the metal alloys. Moreover, what is important from the rheological point of view, an increase of the resting time causes an increase of the yield stress value. This effect results from greater amount of solid particles connections in the alloy structure. It is inseparably linked with the thixotropic transition, reversible isothermal transformation of the gel (having dendritic structure) into the sol (having globular structure). As a result of this transformation the three-dimensional solid skeleton of the gel structure is broken down, which causes decrease of viscosity. Cessation of the material deformation causes rebuilding of the

solid skeleton, what may take place over a considerable time. If this time is longer the solid skeleton became more strong. The figure 10 shows that the yield stress value will equal zero if the shearing process is not stopped. The results of this work prove that the yield point occurs in the semi-solid metal alloys. Similar results were also obtained by other researchers, mainly from RWTH Aachen [13].

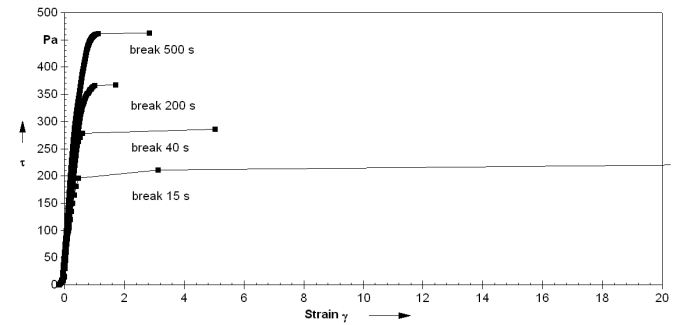


Fig. 9. Flow stress curves for different resting times at 570°C (“break” means resting time)

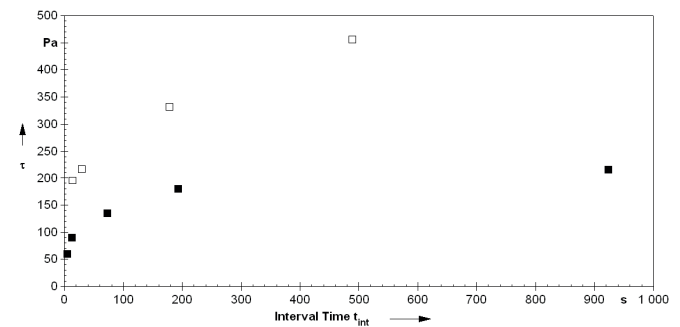


Fig. 10. Values of yield stress versus resting time for samples at: 570°C – white squares, 575°C – black squares

5. Conclusions

During experimental work the shear thinning behaviour of analysed A380.0 aluminium alloy in the semi-solid state was confirmed. Measurements of the viscosity versus the shear rate for different temperatures could be used for development of power cut-off model describing of the alloy rheology in the semi-solid state. This simple model is widely used for simulation of thixocasting processes in software developed for simulation of casting technologies, for example in ProCAST package.

The research methodology allows to identify the thixotropic properties of analysed A380.0 alloy. This behavior was observed on the basis of viscosity measurement versus time for constant shear rate. Moreover, this effect was confirmed by registered values of the yield stress for different values of the resting time.

All measurements were performed using the rotational viscometer with a stationary external cup equipped with the high temperature furnace. The experimental work required application of tools made from special materials. In this work alumina was used because of resistance for the high temperature and low wettability.

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REFERENCES

- [1] D. Weiss, M. Black, The Use of High Pressure Direct Squeeze Casting for Semi-Solid Processing of Aluminum Base Nano Composites. *Solid State Phenomena, Semi-Solid Processing of Alloys and Composites XII*, 72-76 (2013).
- [2] Ł. Rogal, J. Dutkiewicz, H.V. Atkinson, L. Lityńska-Dobrzyńska, T. Czeppe, M. Modigell, Characterization of semi-solid processing of aluminum alloy 7075 with Sc and Zr additions. *Material Science and Engineering A* **580**, 362–373 (2013).
- [3] J. Dutkiewicz, Ł. Rogal, K. Solek, A. Mitura, Thixoforming technology of high carbon X210CrW12 steel. *Int. J. Mater. Form.* **2**, 753-756 (2009).
- [4] K. Solek, T. Stuczynski, A. Bialobrzewski, R. Kuziak, Z. Mitura, Modelling thixocasting with precise accounting of moving front of material, *Mater. Sci. Tech.-Lond.* **21** (5), 551-558 (2005).
- [5] U.A. Curle, J.D. Wilkins, G. Govender, R-HPDC of Magnesium Alloys. *Solid State Phenomena, Semi-Solid Processing of Alloys and Composites XII*, 225-230 (2013).
- [6] K. Solek, A. Bialobrzewski, Modelling of thixocasting processes of magnesium alloys. *Archives of Metallurgy and Materials* **49**, 681-698 (2004).
- [7] H.V. Atkinson, A. Rassili, A review of the semi-solid processing of steel, *Int. J. Mater. Form.* **3**, 791– 795 (2010).
- [8] Metal alloys database, www.totalmateria.com, 2015.
- [9] M. Dziubiński, T. Kiljański, J. Sęk, *Podstawy reologii i reometrii płynów*. Wydawnictwo Politechniki Łódzkiej, Łódź (2009).
- [10] T. Kiljański, M. Dziubański, J. Sęk, K. Antosik, Wykorzystanie pomiarów właściwości reologicznych płynów w praktyce inżynierskiej. Wydawnictwo EKMA Krzysztof Antosik, Warszawa, 9-17 (2009).
- [11] G.T. Mezger, *The Rheology Handbook: For users of rotational and oscillatory rheometers*. 2nd revised edition, Vincentz Network, Hannover, 171-198 (2006).
- [12] H.A. Barnes, Thixotropy - a review, *J. Non-Newtonian Fluid Mech.* **70**, 1-33 (1997).
- [13] A. Moll, M. Modigell, Yield stress phenomena in semi-solid alloys. *Int. J. Mater. Form.* **3**, 779– 782 (2010).

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