Volume 57

O F

M E T A L L U R G Y

DOI: 10.2478/v10172-012-0107-3

M. KOROLCZUK-HEJNAK*, P. MIGAS*

ANALYSIS OF SELECTED LIQUID STEEL VISCOSITY

ANALIZA LEPKOŚCI WYBRANYCH GATUNKÓW STALI W STANIE CIEKŁYM

This paper presents the results of the rheological analysis of selected grades of steel: 90CrV6, DHQ3, 34CrNiMo. In metallurgical processes, gradient of the dynamic viscosity parameter is an important indicator characterizing the behavior of liquid metal in the industrial aggregates. It affects the processes of heat exchange and mass transport occurring in the existing liquid, solid and gaseous phases. Only a small number of high temperature viscosity measurements is available. This is due to the fact that the experiments are difficult to conduct and due to the general assumption that the molten steel is a liquid exhibiting similarities to Newtonian liquids body.

In general liquid metal processes are affected by dynamic forces. Values of the parameters which could be treated as rheological in those real processes are very difficult to measure therefore the influence of the following factors: time of shearing, force value, force direction and shear rate are neglected.

The significance of these dynamic parameters seems to be particularly important in the steel continuous casting and thixoforming process. In this work authors analyzed, from the rheological point of view, chosen representative types of steel produced in electric arc furnace. Measurements were taken using a high temperature viscometer FRS1600 in the range of liquidus temperatures as well as above the liquidus for variable shear rates and different time of shearing.

Keywords: rheology, viscosity, viscometer, liquid steel

W pracy przedstawiono wyniki badań reologicznych wybranych gatunków stali: 90CrV6, DHQ3, 34CrNiMo. W procesach metalurgicznych gradient parametru lepkości dynamicznej jest ważnym wskaźnikiem charakteryzującym zachowanie ciekłego metalu w agregatach przemysłowych. Wpływa on na procesy wymiany ciepła i masy występujące w istniejących fazach ciekłych, stałych i gazowych. Ze względu na skalę trudności eksperymentów wykonuje się niewiele wysokotemperaturowych pomiarów współczynnika lepkości, a rzadko wykorzystuje się parametry reologiczne do jej opisu. Wynika to z powszechnego założenia, że ciekła stal w całym zakresie oddziaływania na nią sił zewnętrznych w trakcie procesu produkcji jest cieczą wykazującą podobieństwo do cieczy newtonowskich.

W procesach przemysłowych ciekły metal poddawany jest działaniu sił dynamicznych (np. oddziaływaniu łuku elektrycznego). Wartości parametrów, które można traktować jako reologiczne w tych procesach rzeczywistych są jak do tej pory bardzo trudno mierzalne. Co za tym idzie, wpływ dynamiki oddziaływania tych czynników: czasu działania siły, jej wartości i kierunku, szybkości ścinania cieczy, na ciekły metal jest zaniedbywany. Znaczenie tych parametrów dynamicznych wydaje się szczególnie ważne w procesie ciągłego odlewania oraz formowania tiksotropowego stali w stanie stało ciekłym. W pracy poddano analizie reologicznej wybrane typowe gatunki stali wytapiane w elektrycznym piecu łukowym. Badania przeprowadzono na reometrze wysokotemperaturowym FRS1600 w zakresie temperatur likwidus , poniżej oraz powyżej tej temperatury dla różnych wartości szybkości ścinania i różnych czasów oddziaływania naprężenia ścinającego na ciekły układ.

1. Introduction

Rheology is the science of flow and deformation due to movements of body particles. Movements of material bodies are connected to the branch of physics known as mechanics. The term rheology originates from a Greek aphorism *panta rei*, "everything flows", it was used for the first time by Eughen Bingham, a professor of the Lehigh University in 1920. Rheology is the study of substances which have a complex molecular structure. In practice, rheology is principally concerned with extending the "classical" disciplines of elasticity and Newtonian fluid mechanics to materials whose mechanical behavior cannot be described with the classical theories [1].

Previous studies of liquid iron solutions were focused on the reliable and accurate measurements of the

^{*} AGH UNIVERSITY OF SCIENCE AND TECHNOLOGY, FACULTY OF METALS ENGINEERING AND INDUSTRIAL COMPUTER SCIENCE, 30-059 KRAKÓW, 30 MICKIEWICZA AV., POLAND

dynamic viscosity coefficient. This value was treated as a physical variable which depends only on temperature and pressure (additionally - on chemical composition). In turn, the research conducted by the authors of this paper more comprehensively defines this coefficient. Existing models, either strictly thermodynamic or semi-empirical, are treating the temperature and chemical composition of the liquid (an iron alloy) as the most important variables affecting the viscosity value. Moreover, existing rheological models are mostly theoretical ones, and were not vet sufficiently verified during high-temperature studies. In most cases, from the real liquid rheological point of view the viscosity coefficient is also dependent on the shear rate, time of shearing and the internal structure. Mass, momentum and energy transport processes in liquid metals will be modeled better if thermo-physical parameters such as density, surface tension, viscosity, diffusivity and thermal conductivity are measured precisely. The accurate measurement of these properties is the key prerequisite to advancement in the development of engineering procedures.

Viscosity is a rheological property of materials which presents itself when the velocity gradient between neighbouring layers of material is observed. Viscosity is an important rheological parameter for understanding the hydrodynamics and kinetics of reactions in metal refining, casting, metal and slag tapping or dripping. For example, the rate of the rise of gas bubbles and non-metallic inclusions through a molten metal is primarily related to viscosity. Also, the kinetics of reactions between metal and slag can be monitored by continuous measurement of the liquids viscosity [2]. From the shear rate to time relationship it is possible to estimate a solid fraction and even solid crystal size at temperatures below liquidus [2]. What is more, viscosity is an indispensable quantity to predict other important transport coefficients, such as diffusivity and thermal conductivity of liquid metals, slags, fluxes [3]. Research on these physical parameters of slags and metals are still conducted.

Rheological characteristics of liquid iron solutions in some range can be compared to the behavior of liquid solutions of iron under the influence of the applied force in time. Such phenomena occur in a real metallurgical processes, e.g. dynamics of the electric arc influence on a liquid steel or dynamic forces during continuous casting process. The values of the parameters, which may be comprehended as rheological parameters in these processes, such as: time of shearing, force value, force direction and shear rate are very difficult to measure. Thus, during the modeling these parameters are not included or included on the basis of theoretical calculations. Most theoretical predictions of transport coefficients of liquid metals are based on the statistical mechanics [2] theory of liquids which assumes that pair interactions are to describe the energy exchange between liquid atoms.

From the rheological point of view we can distinguish three types of a perfect body: St. Veinant, Hookes, Newtonian. The liquid iron solution is widely recognized as a Newtonian perfect body.

Newton was the first who assumed that the resistance of the liquid during the flow is proportional to the relative velocity of the particles (Equation 1):

$$\eta = \frac{\tau}{\dot{\gamma}},\tag{1}$$

where:

- η the coefficient of viscosity, namely the dynamic viscosity (absolute viscosity [Reiner]), Pas,
- τ shear stress, Pa,
- $\dot{\gamma}$ shear rate, s⁻¹

The really existing liquids cannot be defined with a single rheomodel, becasue these fluids, depending on the rheological parameters, might shown similarities to the different perfect body models, thus they are treated as rheologically more complex flows. Figure 1 shows chosen flow curves.



Fig. 1. Illustration of flow principles [4]

Behaviour of liquids without shear yield are presented by curves 1-3:

- 1) flow curve of Newtonian liquid,
- 2) flow curve of shear thinning liquid shear stress growth is slower than the shear rate growth,
- 3) flow curve of shear thickening liquid shear stress growth is faster than the shear rate growth.

Properties of liquid with shear yield are presented by curves 4-6:

- 4) flow curve of Bingham plastic behaviour,
- 5), 6) flow curve of pseudo-plastic behaviour.

Thanks to rheological tests on a high-temperature rheometer it is possible, by observing changes in the viscosity values for the measured parameters (temperature, shear rate, shear time), to investigate the structural changes occurring in the liquid and semi – solid iron solutions.

Previous studies of liquid metal solutions were focused on the accurate measurement of the viscosity values under the given conditions of the experiment. The authors of this paper are planning to use the rheological measurements to study the rheological nature of the liquids.

In the past, several theories have been put forward to explain the viscosity of liquid metals and alloys. Among the most fundamental ones are those of Born & Green, Rice & Kirkwood and Rice & Alnatt [5]. Authors [6] propose a hypothesis of holes or voids in the liquid metal alloys into which atoms can jump as a major mechanism of the viscous flow. Guthrie [6] points out this hypothesis, which is based on an extrapolation of solid structures remains unsubstantiated due to the lack of experimental evidence of actual voids in a melt. The next theory has been based on the potential between atom pairs.

In general, researchers tend to use rather semi-empirical models. However, such simple models have no general acceptance in literature since their applicability changes from case to case [5].

There are many semi-empirical equations to predict the relation between temperature and viscosity of metals at their melting points. Estimating the viscosity of a partially solidified metals and alloys is an important problem in structural analysis of casting and thixoforming processes.

Models used in literature [7] allow to predict viscosity value of liquid metal alloys (previously they have been used mainly to calculate the viscosity of low-melting metals like aluminum, zinc, lead, tin, copper and antimony) using thermodynamic properties such as molar enthalpy of mixing, molar free energy and physical properties such as viscosity of the components, density, molar volume of alloys, atomic mass and atomic radius. These models are largely theoretical models which do not take into account any rheological parameters describing flow of materials such as force (shear rate, rotation speed) and time (shear time). Moreover, it is not known if these models work properly to predict viscosity of non-Newtonian fluids and how they work if we were to change the temperature, chemical composition of alloys and any of the rheological parameters.

2. Experimental method

Viscosity measurement using different values of the rheological parameters (shear rate, shear time) were conducted for three steel grades: 90CrV6, DHQ3, 34CrNi-Mo in the range of 1440 to 1510°C. Chemical compositions of steel were shown in Table 1.

Chemical compositions of investigated steel

С	Mn	Si	Р	S	Cu	Cr	Ni	Мо	V	
%										
90CrV6										
0.89	0.26	0.19	0.008	0.005	0.08	1.43	0.44	0.06	0.106	
34CrNiMo										
0.39	0.62	0.24	0.010	0.005	0.09	1.57	1.67	0.26	0.074	
DHQ3										
0.80	0.26	0.69	0.010	0.004	0.07	2.93	0.13	0.54	0.011	

Studies were carried out by using the high temperature rheometer FRS1600 which was designed as a result of cooperation of Anton Paar company and the Faculty of Metals Engineering and Industrial Computer Science at the AGH University of Science and Technology. Rheometer equipped with concentric cylinder measuring system works in accordance with the Searle's method [8] (with motion bob) (Fig. 2). Measuring system: bob and crucible were made of alumina with addition of zirconium oxide.



Fig. 2. Equipment and measuring system

The liquidus point for each steel was calculated using ProCAST software and Loesser's and Wansel's tables [9]. The liquidus temperature for the investigated steels

TABLE 1

were: 1463°C for 90CrV6, 1458°C for DHQ3, 1490°C for 34CrNiMo.

Tests were carried out according to the measuring scheme presented in table 2. This scheme was applied to each of the investigated steel grades at five temperatures. The paper presents the selected results of a rheological analysis at two temperatures: liquidus point, and 20 degrees below and over the liquidus temperature.

Temperature measurement accuracy is ± 5 degree. The experiment varies the temperature slightly. However before the measurement calibration of the measuring system is performed from the viewpoint of those changes.

Measuring scheme of investigated steel in the FRS 1600 rheometer

Rotation speed [rpm] 2 5 10 15 20 Time [s] 15 15 15 15 15 Rotation speed [rpm] 20 15 10 5 2	1 20
	20
Rotation speed [rpm] 20 15 10 5 2	
Time [s] 60 60 60 60 60	
Rotation speed [rpm] 30 1 30 1	
Time [s] 40 30 40 60	
Rotation speed [rpm] 12 1 12 1	
Time [s] 40 30 40 60	

Applied shear rates are selected according to geometry of the used measuring system and range of viscosity values, in order to assure laminar flow between two parallel layers of fluid during simple shearing. This follows from the definition of viscous flow.

3. Investigation and analysis of the results

Authors of [10] studied the rheological behaviour from the point of view of potential application in thixoforming. Due to that fact, they used large values of shear rate. In the investigated semi-liquid system the solid fraction was taken into account.

Authors of this paper focus on iron solutions in the range of liquidus temperature for different grades of steel. Applied values of the temperature and shear rate were based on real metallurgical processes.



Fig. 3. Shear stress vs. shear rate for steel X210CrW12 at the liquidus temperature [10]

Figure 4 [10] shows the shear stress as a function of shear rate for completely (100%) liquid material. Value of shear rate strongly affects the shear stress and thus the proportionality factor is viscosity.



TABLE 2

Fig. 4. Changes of shear stress vs. shear rate at the liquid temperature

The results obtained for shear rate in the range of $0.35-5.23s^{-1}$ (Fig. 3) for analyzed iron solutions coincide with those of the authors [10]. Based on the results of the rheological tests in the present work, the observed influence of shear rate on shear stress value, was similar to that perceived by the authors of [10], however in this case the value of shear rate was much lower. Grades of steel with higher content of chromium and silicon tend to display a higher value of shear stress for the same value of shear rate (DHQ3, 34CrNiMo, 90CrV6 – adequately).

Trend lines showing the changes of the viscosity have been plotted for the highest regression coefficient, after a preliminary statistical analysis.

Figure 5 presents the results of changes of the dynamic viscosity coefficient in time, for five different values of rotation speed: 2, 5, 10, 15, 20 rpm, which corresponds to the following values of shear rates: 0.35, 0.87, 1.74, 2.62, $3.49s^{-1}$. Changes of shear rate were made every 15 seconds, the experimental temperatures were 20 Celsius degrees below the liquidus temperature for each of the studied steels. An influence of the dynamic viscosity of liquid metal on shear rate is visible, especially for the low shear rate values. The effect of time on the change of the viscosity at constant value of $\dot{\gamma} =$ $0.35s^{-1}$ for first 15seconds is also visible. The value of the dynamic viscosity decreases dramatically. In the next interval the value of viscosity for shear rate $\dot{\gamma} = 0.87s^{-1}$ still decreases, but the changes are not so spectacular. For shear rate over 1.74 s⁻¹ (10 rpm) viscosity decrease in time is negligibly small.



Fig. 5. Characteristics of viscosity vs. shear rate in time (at temperature 20°C below the liquidus point)



Fig. 6. Characteristics of viscosity vs. shear rate in time (at temperature of liquidus)

Figure 6 shows the results of the rheological measurements for the same scheme as in the Fig. 5. The theoretical point of liquidus temperatures in this experiment was calculated for all investigated grades of steel.

Coefficient of the dynamic viscosity depends significantly on shear rate (for low value) and on time of shearing, although no big differences were observed between the viscosity value of fully liquid steel and in temperatures 20 Celsius degrees below liquidus point.

Character of changes of the dynamic viscosity coefficient in Fig. 6 is very similar to that which was presented for measurement conducted at 20 degrees below the liquidus temperature. Comparing the obtained values no significant differences in values of the viscosity were observed.

Figure 7 presents changes of the dynamic viscosity coefficient value for selected grades of steel for shear rate: $\dot{\gamma} = 1.74 \text{s}^{-1}$, $\dot{\gamma} = 2.62 \text{s}^{-1}$, $\dot{\gamma} = 3.49 \text{s}^{-1}$.



Fig. 7. Change of the viscosity vs. time, in temperature 20°C over the liquidus point



Fig. 8. Change of the viscosity vs. time, in temperature 20°C below the liquidus point

Figure 7 presents changes of the viscosity value in time for different values of shear rate. The temperature during the experiments was 20°C over the liquidus point. The difference of the dynamic viscosity coefficient values is visible for different grades of steel, however this difference is small. Investigated grades of steel are fully presenting the nature of Newtonian liquids, for three applied values of shear rates.

Figure 8 presents changes of the viscosity value in time for different values of shear rate. The temperature during the experiments was 20° C below the liquidus point. The nature of the plots is very similar to the plots created for liquidus temperature. A slight increase of the viscosity values was observed in the second interval (40-70 seconds) in comparison to this in graph 7, while the value of the viscosity, for shear rate equals $5.23s^{-1}$, and does not differ for both Figures (7 and 8).

4. Conclusions

The following can be stated based on the results of the rheological analysis of liquid steel 90CrV6, DHQ3, 34CrNiMo:

- for such small range between liquidus temperature and 20°C below, there were no significant differences in the values of the dynamic viscosity coefficient of the analyzed steel; it may be a very small share of the solid phase on these measurement systems or this phase has little effect on the viscosity of these systems,

– analyzed grades of steel in the investigated temperatures for the assumed low value of shear rate are non-Newtonian fluids; they show the shear-thinning properties, while for the shear rate above $5.23s^{-1}$ they show the nature of Newtonian liquid body,

-20 degrees over the liquidus temperature investigated grades of steel show similarity to the ideal Newtonian body, in the whole range of applied shear rate,

– for low value of shear rate, the investigated steel shows time-dependent properties, the increase of the viscosity is not abrupt, but it varies regularly in time during shearing on the liquid medium,

– the influence of the chemical composition on the dynamic viscosity coefficient value of liquid systems is clearly visible, though the rheological characteristics of the analyzed steels are similar. However, the impact of the individual elements on the viscosity value cannot be clearly determined from the obtained results. It should be noted that the performed experiments gave interesting results therefore we will continue them in the future. The authors predict a continuation of the rheological analysis of liquid solutions of iron with the use of other geometry of measurement systems, including a narrow gap system. The authors also plan experiments with solid particles imitating precipitates in high temperature solutions.

Acknowledgements

Financial support of National Science Center for research project "Developing an empirical model of the rheological properties of liquid metals on the example of iron sulutions" no. 2011/01/N/ST8/07368 is gratefully acknowledged.

REFERENCES

- J. Ferguson, Z. Kembłowski, Reologia stosowana płynów, Wydawnictwo MARCUS, Łódź 1995.
- [2] S.I. Bakhtiyarov, R.A. Overfelt, Measurement of liquid metal viscosity by rotational technique, Acta Metallurgica Inc. 47, 17, 4311-4319 (1999).
- [3] P. Migas, M. Karbowniczek, Interactions between liquid slag and graphite during the reduction of metallic oxides, Archives of Metallurgy and Materials, Polish Academy of Sciences, Committee of Metallurgy, Institute of Metallurgy and Materials Science 55, 1147-1157 (2010).
- [4] Praca zbiorowa pod redakcją Z. Kembłowskiego, T. Kiljańskiego, Ćwiczenia laboratoryjne z reometrii technicznej, Skrypty dla Szkół Wyższych Politechnika Łódzka, 18-24, Łódź 1993.
- [5] P. Terzieff, Review. The viscosity of liquid alloys, Journal of Alloys and Compounds **453**, 233-240 (2008).
- [6] S. S r i d h a r, Estimation models for molten slag and alloy viscosities, JOM **54**, 11, 46-50 November 2002.
- [7] M. Kucharski, Lepkość roztworów metali, stopionych soli o wspólnym anionie żużlowym, Zeszyty Naukowe Akademii Górniczo-Hutniczej im. Stanisława Staszica, Zeszyt 128, Kraków 1989.
- [8] T.G. M e z g e r, The rheology handbook. For users of rotational and oscillatory rheometers. 2nd revised edition, Vincentz Network GmbH & Co. KG, Hannover, 171-172 (2006).
- [9] E. S c h u r m a n n, T. S t i s o v i c, Stahl u. Eisen 118, 11, 97-102 (1998).
- [10] Edited by G. Hirt, R. Kopp, Thixoforming. Semi-solid Metal Processing, WILEY-VCH Verlag GmbH & Co. KGaA, 183-186, Wienheim (2009).