

M. KOROLCZUK-HEJNAK*, P. MIGAS*

SELECTED GRADES OF STEEL AS RHEOLOGICALLY DEFINED LIQUID BODIES

WYBRANE GATUNKI STALI JAKO CIECZE DEFINIOWANE REOLOGICZNIE

In many processes liquid metal is subjected to dynamic forces, e.g. dynamics of the electric arc, dripping of pig iron in a blast furnace in counter current with reducing gas, in a continuous casting process - dynamic phenomena during flow and crystallization. Values of rheological parameters in these real processes are, so far, very difficult to measure. And thus the influence of the dynamics of these factors: liquid shear rate, time of force action, its value and direction are neglected. In this work the authors analyzed, from the rheological point of view, some selected typical types of steel: 90CrV6, DHQ3, 34CrNiMo, produced in an electric arc furnace. Measurements were taken using a high temperature rheometer FRS1600 in the range of liquid and under-liquid temperatures for changing shear rates and different times of shear stress impact on the liquid system. The measured rheological characteristics allowed us to conclude that the investigated steels show relationships between shear rate and time when the liquid steel was under the influence of the dynamic forces.

Keywords: rheology, viscosity, liquid steel

W wielu procesach ciekły metal poddawany jest działaniu siły dynamicznej, m.in. dynamiki działania łuku elektrycznego, ściekania surówki w wielkim piecu w przeciwwądzie z gazem redukcyjnym, COS – zjawisk dynamicznych w trakcie przepływu i krystalizacji. Wartości parametrów, które można traktować jako reologiczne w tych procesach rzeczywistych są jak do tej pory bardzo trudno mierzalne. A co za tym idzie, wpływ dynamiki działania tych czynników: szybkości ścinania cieczy, czasu działania siły, jej wartości i kierunku jest zaniedbywany.

Poddano analizie reologicznej wybrane typowe gatunki stali: 90CrV6, DHQ3, 34CrNiMo, wytapiane w elektrycznym piecu łukowym. Badania przeprowadzono na reometrze wysokotemperaturowym FRS1600 w zakresie temperatur likwidus i poniżej likwidus dla różnych wartości szybkości ścinania i różnych czasów oddziaływania naprężenia ścinającego na ciekły układ. Wykonano charakterystyki reologiczne, które pozwalają stwierdzić, że analizowane stale wykazują zależności od szybkości ścinania i czasu, w którym poddajemy ciekłą stal działaniu sił dynamicznych.

1. Introduction

Mathematical modeling and the control of molten metal processing operations require the knowledge of the thermo-physical properties of liquid metals at their melting points. Mass, momentum and energy transport processes in liquid metals will be well understood 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 an advancement in the development of engineering procedures.

In most of metallurgical processes liquid iron solutions flow freely, or in some cases the behaviour of liquid iron solutions is influenced by the applied force

and time. Such phenomena occur in real metallurgical processes, e.g. dynamics of the electric arc influencing liquid steel, pig iron in a blast furnace dripping in counter-current with reducing gas and during the continuous casting process. The values of the parameters such as duration of the force, force value and direction, shear rate are very difficult to measure.

Rheology is the science of flow and deformation due to the movements body in time. Study of movements of material bodies forms part of the science of mechanics. Rheology is the study of flow of substances which have complex molecular structures; in practice it is principally concerned with extending the "classical" disciplines of elasticity and (Newtonian) fluid mechanics to materials whose mechanical behaviour cannot be described with classical theories [1].

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

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

$$\eta = \frac{\tau}{\dot{\gamma}}, \quad (1)$$

where:

- η – the coefficient of viscosity, namely the dynamic viscosity [Pas],
- τ – shear stress [Pa],
- $\dot{\gamma}$ – shear rate [s^{-1}].

Viscosity is a rheological property of materials when the velocity gradient between different layers of material is present. Viscosity is an important rheological parameter for understanding the hydrodynamics and kinetics of reactions in metal 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 its viscosity. Also, the kinetics of reactions between metal and slag can be determined by continuous measurement of the liquid's viscosity [2]. From the relationship between shear rate and time, it is possible to estimate a solid fraction and even solid crystal size at temperatures below those of melting [2]. What is more, viscosity is the indispensable quantity to predict other important transport coefficients, such as diffusivity, thermal conductivity and surface tension of liquid metals, slags, fluxes [3]. Research relating to these physical parameters is still being conducted.

M. Modigell et. al [7] carried out a rheological analysis of steel X210CrW12 from a thixoforming point of view, using a large value of shear rate and taking into account the solid fraction. The applied values of temperature and shear rate were based on real metallurgical processes. The results of these tests shed light on the changes in the dynamic viscosity coefficient at high shear rates reaching approx. $250 s^{-1}$. Such shear rates are present in molten metal processing. These results suggest that the dynamic viscosity of steel in semi-solid state depends to a large extent on shear rate. The authors claim that steel X210CrW12 in liquid state has characteristics of a Newtonian fluid.

The following paper presents the research into the character of changes in the viscosity coefficient for selected grades of steel: DHQ3, 34CrNiMo, 90CrV6.

2. Theoretical points of measurements of dynamic viscosity coefficients

Most theoretical predictions of transport coefficients of liquid metals are based on the mechanico-statistical theory of liquids which assumes that pair interactions are

primary interaction forces and are sufficient to describe the energy exchanges between liquid atoms.

In the past, several theories have been put forward to explain the viscosity of liquid metals and alloys [4]. In general, experimentalists tend to use rather semi-empirical models instead of the full theory which requires more theoretical skill. However, such simple models have no general acceptance in literature, since their applicability changes from case to case [4].

There are many semi-empirical equations to predict temperature dependences of the viscosity of metals near their melting points. Estimating the viscosity of partially solidified metals and alloys is an important problem in the structural analysis of casting and thixoforming processes. Models used in the literature [5] allow us to predict the viscosity of liquid metal alloys. These models have been previously used mainly to calculate the viscosity of low-melting metals as aluminum, zinc, lead, tin, copper and antimony using thermodynamic properties such as molar enthalpy of mixing, free molar energy and physical properties such as the viscosity of the components, density, the molar volume of alloys, atomic mass and atomic radius. These models are largely theoretical and do not take into account rheological parameters describing the flow of materials, such as force, shear rate, and time. Moreover, it is not known if these models can accurately predict viscosity of non-Newtonian fluids and the effects of change of temperature and chemical composition of the alloys. However, they allow us to predict the viscosity of multicomponent systems by obtaining the thermodynamic value for 'pure' (mono) components.

This approach to the theoretical modeling of the dynamic viscosity coefficient of liquid iron solutions stems from the complexity of the experiment and the measuring equipment which needs to be constructed. The basic difficulties arise from the high temperature at which the measurements are carried out and from the corrosive action of the sample on the materials of the measuring system. First of all, materials used in the measurements must ensure the stability of linear dimensions over time in very extreme conditions – high temperature and contact with liquid iron. The methodology of the experimental procedure (which should provide the correct rheological data) is probably the main difficulty, because it is still not well developed.

Currently literature containing reliable data values of the viscosity coefficient of liquid steel is very limited, and hardly ever it takes into account the changes of the rheological parameters.

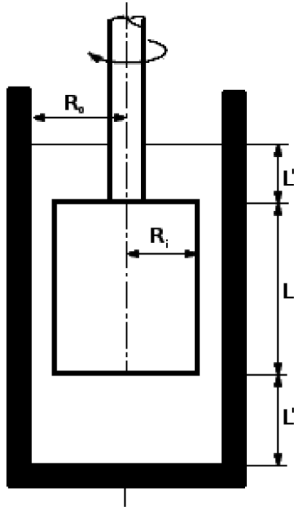


Fig. 1. Concentric cylinder measuring system [6]

Figure 1 presents the most important linear dimensions of the measurement system used for rheological research into liquid iron solutions.

When measuring viscosity using the concentric cylinder method, the rheological parameters are related to the radii of these cylinders. Shear stress is defined as:

$$\tau(r) = \frac{M}{2\pi L r^2}, \quad (2)$$

where:

- M [Nm] – the torque on the surface of the rod,
- L [m] – the length of the cylindrical part of the rod,
- r [m] – the radius (the distance between the rotation axis and any layer of the liquid).

$R_i \leq r \leq R_e$ and the shear stress can be expressed as:

$$\tau(r) = \frac{M}{2\pi L R_i^2}, \quad (3)$$

In the same “type” of measuring system shear rate is defined by the formula:

$$\dot{\gamma}(r) = \frac{1}{r^2} \frac{2R_e^2 R_i^2}{R_e^2 - R_i^2} \omega, \quad (4)$$

where:

- R_i – value of the inner radius [m],
- R_e – value of the outer radius [m],
- ω – angular speed [rad/s], $\omega = (2\pi \cdot n) / 60$,
- r – distance from the rotational axis [m],
- n – rotational speed – rpm [1/min or min^{-1}].

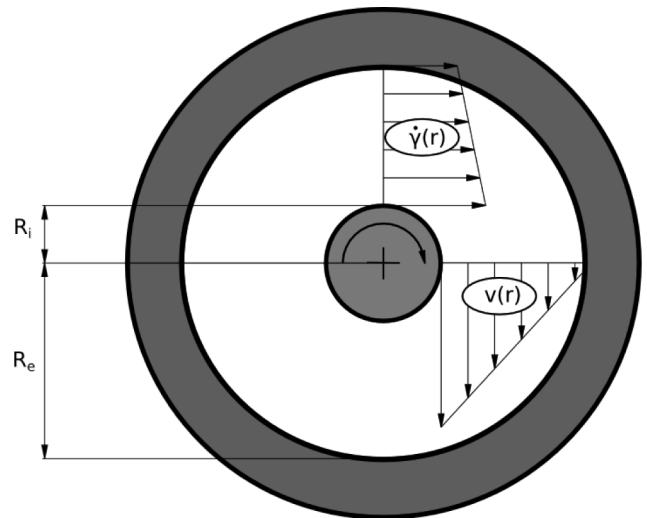


Fig. 2. Cross-section of a concentric cylinder measuring system [6]

The distribution function of the circumferential velocity in the shear gap is:

$$v = \omega \cdot r, \quad (5)$$

and the shear rate:

$$\dot{\gamma}(r) = \left[\frac{(2R_e^2)}{R_e^2 - R_i^2} \right] \cdot \omega, \quad (6)$$

For $r = R_i$:

$$\eta = \frac{\tau(r)}{\dot{\gamma}(r)} = \frac{\tau(R_i)}{\dot{\gamma}(r)} = \left(\frac{R_e^2 - R_i^2}{4\pi L R_e^2 R_i^2} \right) \cdot \frac{M}{\omega}, \quad (7)$$

The above formulae (2-7) and the analysis of the experimental data obtained in low temperature conditions show that it is necessary to choose the appropriate geometry (and linear dimensions) of the measuring system for the experimental parameters of liquid iron alloys (especially liquid steel) [6].

3. Experimental procedures

Viscosity measurements using different values of the rheological parameters were conducted for three chemical compositions: 90CrV6, DHQ3, 34CrNiMo. Chemical compositions of the steels are shown in Table 1.

Chemical compositions of the investigated steels

Grade of steel/Chemical composition%	C	Mn	Si	P	S	Cu	Cr	Ni	Mo	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

Liquidus temperatures were calculated for all of the investigated grades of steel using ProCAST software and Loessera and Wansela tables – commonly used in industry. These were: 1463°C for 90CrV6, 1458°C for DHQ3, 1490°C for 34CrNiMo. Measurements were made at the calculated liquidus temperature (assuming that the solution is totally liquid) and at 40°C below this temperature (basically about 10% of solid fraction). Liquidus temperature value is a boundary condition for providing the rheological measurements in different physical states (liquid, semi-solid).

Studies were carried out using the high temperature rheometer FRS1600. This rheometer, a concentric cylinder measuring system, works as in Searle’s method – Fig. 1. Measuring system: bob and crucible were made of alumina oxide with small amount of zirconium oxide. Tests were conducted with both smooth bob and crucible with a wide rheological gap of 7.5mm.

Table 2 presents the measurement schemas, according to which the measurements were conducted. After

reaching the desired temperature, 10 minutes of measurement was conducted at a bob speed of 1 rpm. Next the bob speed was increased, from 2 rpm to 20 rpm every 3 minutes. In the second set of experiments the measuring bob was set initially to rotate at 1 rpm for 3 minutes, then was accelerated to 30 rpm. Then the speed was reduced to 1 rpm for 3 minutes and was accelerated to 30 rpm for 10 minutes. These cycles were repeated for each temperature and for each steel grade.

The schemas depend on the continuous changes of spindle (bob) speed, related to the shear rate for each measurement. Dynamic viscosity coefficient was recorded at different frequencies, each corresponding to a single measurement point. Thus, the measurement points are characterized by a variable duration.

In order to homogenize the temperature of the sample, mixing took place for 10 minutes without any data being recorded. After that, the measurement schema was carried out.

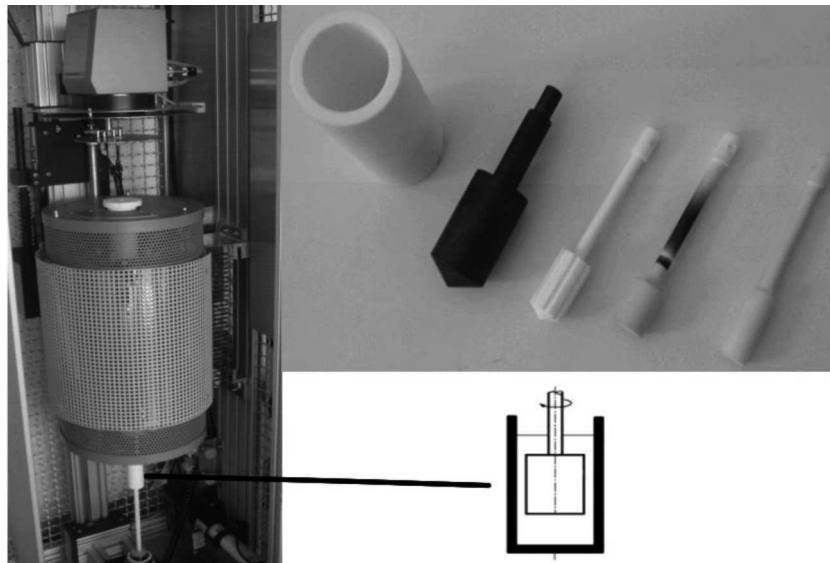


Fig. 3. Equipment and elements of measuring system

Measuring scheme of investigated steels in the rheometer FRS 1600

I	Rotation speed [rpm]	1	2	5	10	15	20
	Shear rate [s⁻¹]	0.17	0.35	0.87	1.74	2.62	3.49
	Time [min]	10	3	3	3	3	3
II	Rotation speed [rpm]	1	30	1	30		
	Shear rate [s⁻¹]	0.17	5.23	0.17	5.23		
	Time [min]	3	10	3	10		

4. Results of the experiments

The Figs (4-8) which are presented below are plotted as the dependence of viscosity and number of measuring points (changes of shear rate). The nature of the plots resulted from the focus of the viscosity changes over time for the constant (in ranges) shear rate. Assuming that the test fluid had Newtonian characteristics – such changes were not expected. In another case (with a non-Newtonian fluid), such changes occur.

Figure 4 presents the results of changes of the dynamic viscosity coefficient in time, for six different values of rotation speed: 1, 2, 5, 10, 15, 20rpm, which correspond to the following values of shear rate: 0.17, 0.35, 0.87, 1.74, 2.62, 3.49 [s⁻¹].

What becomes clear are the differences in the dynamic viscosity rate depending on the shear rate. The values of dynamic viscosity rate with low shear rate for

steel DHQ3 are the smallest. For the value of $\dot{\gamma} = 0.17\text{s}^{-1}$ the dynamic viscosity rate has a value at the level of 0.08 Pas. The remaining two constantly reach the value of approx. 0.25 Pas. The differences for the shear rate of 0.35 and 0.87 are similar. For the shear rate of 1.74 – 3.49s⁻¹ the differences are not so clear anymore. With a higher rotational speed of the bob the viscosity values do not change dramatically for other investigated grades of steel, either. It can mean that the chemical composition of investigated steel affects the values of viscosity in the case of lower shear rate rather than the shear rate higher than 1.74 s⁻¹.

Figures 5a, 5b, 5c show the results of the rheological measurements for the same scheme as in Fig. 4.

Changes of shear rate were made every 3 minutes (10 minutes for 1 rpm), the measurement temperatures were 40 Celsius degrees below the liquidus temperature for each of the studied grades of steel.

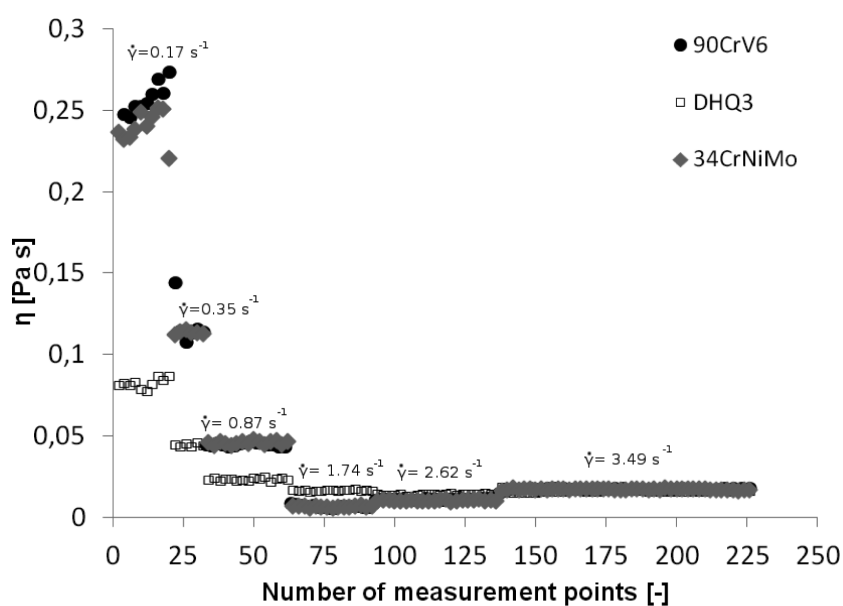


Fig. 4. Characteristics of viscosity vs. number of measurement points. Liquid temp

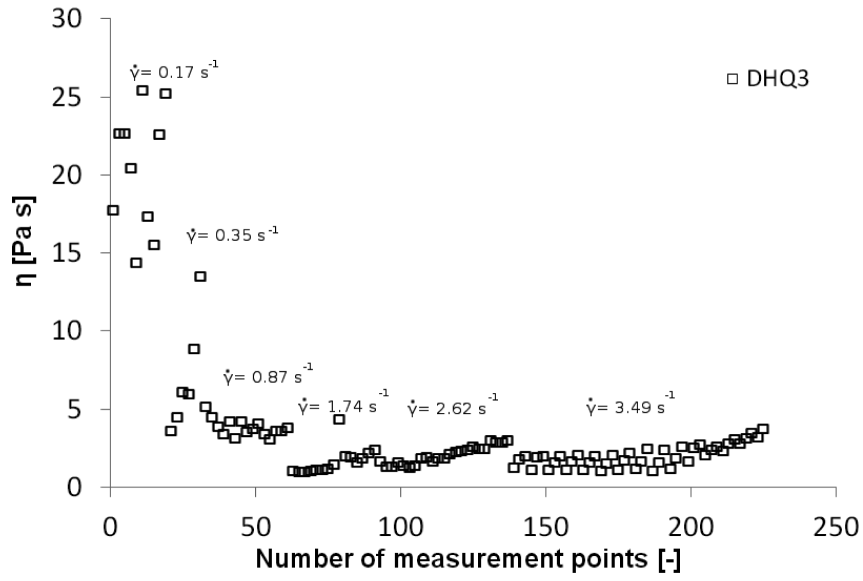


Fig. 5a. Characteristics of viscosity vs number of measurement points. Below liquid temp

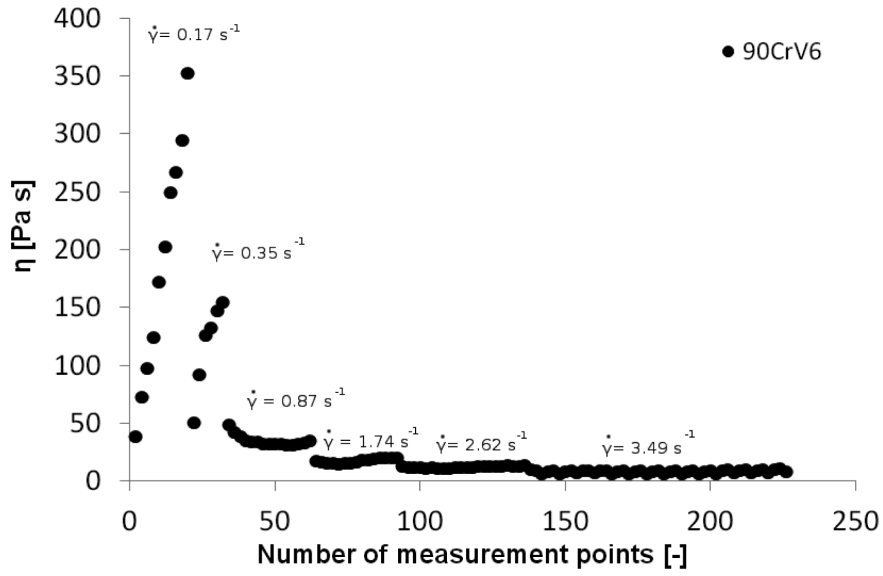


Fig. 5b. Characteristics of viscosity vs number of measurement points. Below liquid temp

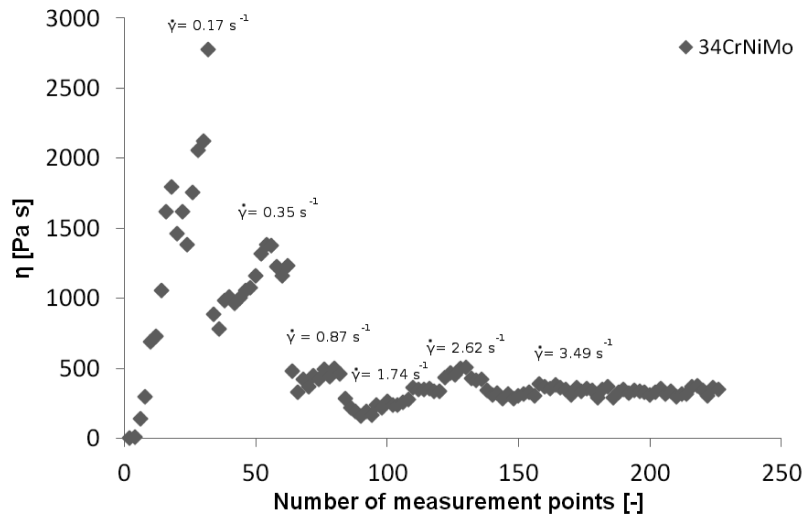


Fig. 5c. Characteristics of viscosity vs number of measurement points. Below liquid temp

The obtained values of dynamic viscosity rate for all three grades of steel in the quenched state (by 40°C) show a dependence on the shear rate. The changes are especially visible for lower shear rates at the level of 0.17 and 0.35. For higher rates 0.87, 1.74, 2.62, 3.49 the changes of η are not so big. It is clear that there are differences in the viscosity values depending on the grade of analyzed steel. Not only the tendency of changes but also the values – η are subject to fluctuations – some even an order of magnitude greater than the values for other grades of steel. The following grades: 34CrNiMo, 90CrV6 DHQ3 had the highest viscosity values within the shear rate of 0.17 to 0.87. The lowest viscosity values were obtained for steel containing the highest amount of carbon and for alloying elements, and so was the case for

the liquidus temperature. An influence of the dynamic viscosity of liquid metal on shear rate is visible, especially for the low shear rate values.

Figure 6 presents the changes of the dynamic viscosity coefficient value for selected grades of steel for two values of shear rate: $\dot{\gamma} = 0.17s^{-1}$ (1rpm) and $\dot{\gamma} = 5.23s^{-1}$ (30rpm). The measurements were conducted in the liquid temperatures, previously calculated for each steel.

For the highest value of shear rate ($5.23s^{-1}$) viscosity shows a linear character at the same level for the corresponding time intervals – 120 s. For the lowest shear rate ($\dot{\gamma} = 0.17s^{-1}$) we can observe differences in steel viscosity values.

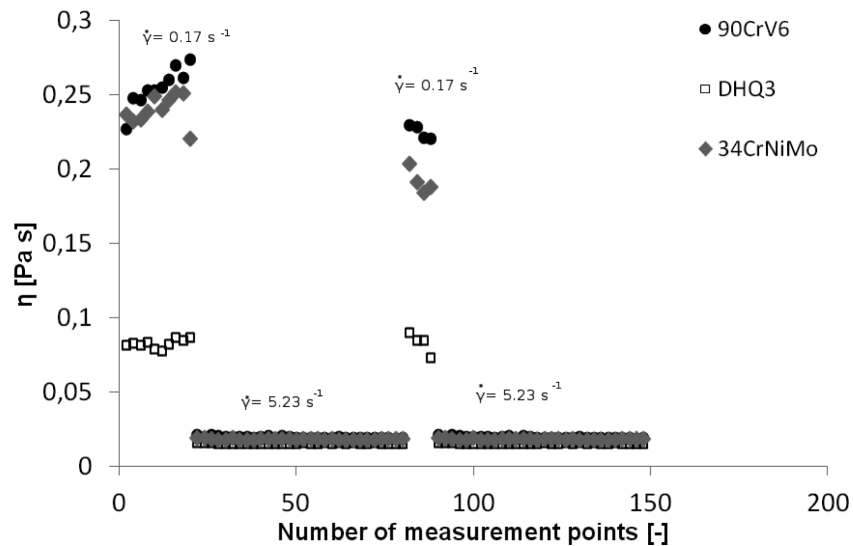


Fig. 6. Characteristics of viscosity vs number of measurement points. Liquid temp

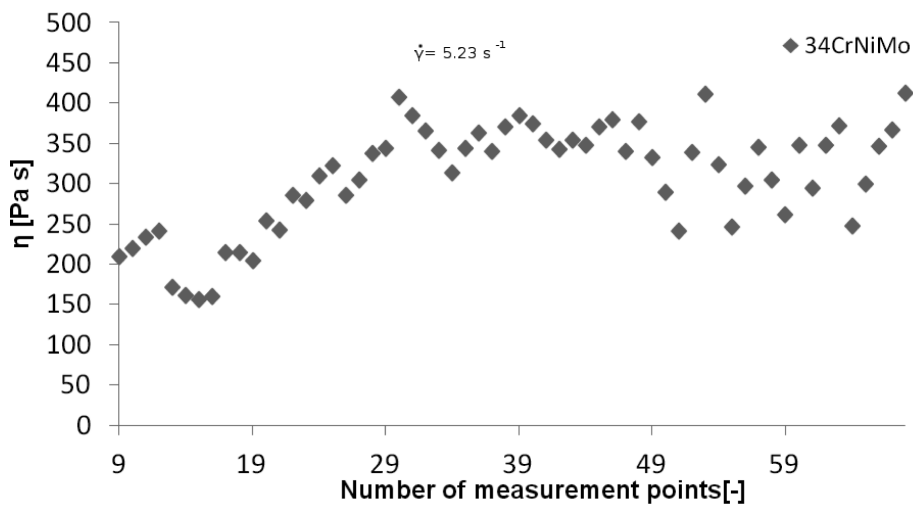


Fig. 7. Characteristics of viscosity vs number of measurement points. Highest shear rate. Below liquid temp

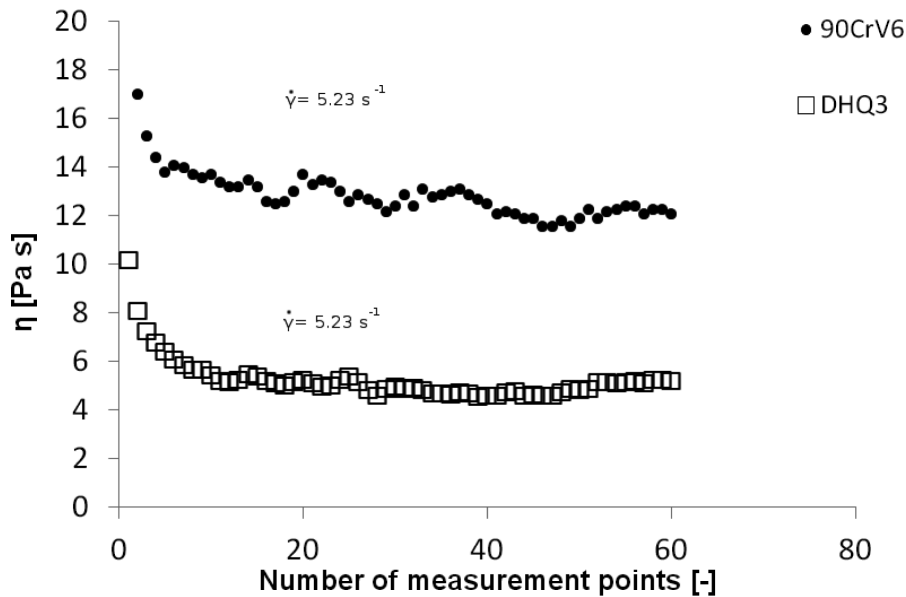


Fig. 8. Characteristics of viscosity vs number of measurement points. Highest shear rate. Below liquid temp

Figures 7 and 8 present the changes in viscosity coefficient in the case of the highest value of shear rate in the temperature 40°C lower than liquidus temperature for the analyzed grades of steel. Quenched steel 34CrNiMo in Fig. 7 shows non-linear changes in viscosity for a constant value of the shear rate. The viscosity reaches high values between 200-400Pas. As a result, it is probable that solid phase extraction takes place in the volume of the liquid in the temperature 40°C lower than the liquidus temperature. These solid phases significantly affect the viscosity values and the character of the changes.

Figure 8 presents the changes in viscosity for the two remaining steel grades in quenched state. What can be observed is a different character of obtained changes and what is more, viscosity values are much lower than for steel 34CrNiMo.

The character of changes of the viscosity coefficient for both steel grades seems to be similar. Steel DHQ3 has lower viscosity values – at the level of 6Pas, while steel 90CrV6 has viscosity values at the level of 12-14Pas. It is possible and probable that crystal extraction occurs but the character, shape, position and placement of these crystals in the volume of the liquid will be totally different than in the case of steel in Fig. 7.

5. Conclusions

The experimental research suggest the following:

- for low shear rates the analyzed iron solutions in liquidus temperatures show variability in viscosity, which depends on applied shear stress; for higher shear rates on the other hand the analyzed solutions show a similarity to Newtonian fluids,

- the viscosity of liquid steel depends on the chemical composition, this dependence is more visible for semi-solid steel than for liquids,
- liquid state steel is characterized by a very low value of dynamic viscosity coefficient.

Credits

These experiments and studies were conducted as part of a research project: „Developing an empirical model of the rheological properties of liquid metals on the example of iron solutions” number 2011/01/N/ST8/07368 financed by the National Science Centre.

REFERENCES

- [1] 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* **55**, 1147-1157 (2010).
- [4] P. Terzieff, Review. The viscosity of liquid alloys, *Journal of Alloys and Compounds* **453**, 233-240 (2008).
- [5] M. Kucharski, *Lepkość roztworów metali, stopionych soli o wspólnym anionie żuźlowym*, Zeszyty Naukowe Akademii Górniczo-Hutniczej im. Stanisława Staszica, Zeszyt 128, Kraków 1989.

- [6] Thomas G. Me z g e r, The Rheology Handbook. For users of rotational and oscillatory rheometers. 2nd revised edition., Vincentz Network, Hannover 2006.
- [7] M. Modigell et al., edited by G. Hirt, R. Kopp, Thixoforming. Semi-solid Metal Processing, WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim, 2009, pp. 167-220.

Received: 10 November 2011.