A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
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AN ANALYSIS OF STEEL VISCOSITY IN THE SOLIDIFICATION TEMPERATURE RANGE

ANALIZA LEPKOŚCI STALI W ZAKRESIE TEMPERATUR KRZEPNIĘCIA

The main objective of this study was to conduct an analysis of the rheological properties of steel in a semi-solid state. The results were used for the development of mathematical models of the apparent viscosity.

Knowledge of the rheological properties is crucial for the numerical modeling of technological processes. Shaping in the semi-solid state, also known as thixoforming processes, is an innovative method of processing metal alloys and has a great many advantages in comparison with classical metal forming and foundry processes.

Nowadays, research is conducted with the practical application of this method in steel processing [1,2] as its goal. The most significant achievement of this particular study is the application of a viscometer which was specially designed for material tests executed at extremely high temperatures, such as the measurement of liquid or semi-liquid steel viscosity. This paper presents the results of a rheological analysis of 100Cr6 (PN ŁH15) tool steel. It was performed using a rotational viscometer with a stationary external cup.

Keywords: thixoforming, rheology, viscosity, rheological models, viscometer

Głównym celem pracy była analiza właściwości reologicznych wybranej stali w stanie stało-ciekłym. Otrzymane wyniki zostały wykorzystane do opracowania modelu matematycznego lepkości pozornej. Znajomość własności reologicznych jest konieczna w numerycznym modelowania procesów kształtowania metali. Przeprowadzona w tym przypadku analiza będzie służyć do modelowania procesów formowania stopów żelaza w stanie stało-ciekłym, nazywanych również procesami formowaniem tiksotropowego. Procesy te biorą swoją nazwę od zjawiska tiksotropii jakie zachodzi w trakcie odkształcenia stało-ciekłego metalu. Zjawisko to przejawia się zależnym od czasu zachowaniem materiału, dla którego lepkość pozorna zmniejsza się wraz ze wzrostem czasu odkształcenia oraz ponownie wzrasta do poprzedniej wartości po jego zaprzestaniu i jest wynikiem transformacji, którą można zdefiniować jako odwracalną przemianę żelu (stop, w którym faza stała występuje w postaci spójnego szkieletu zanurzonego w fazie ciekłej) w zol (stop posiadający strukturę globularną), zachodzącą pod wpływem oddziaływania mechanicznego (mieszanie, wstrząsy). W rezultacie następuje zniszczenie przestrzennej sieci żelu, co powoduje jego upłynnienie (spadek lepkości pozornej), czyli przejście w zol. Skutkiem formowania tiksotropowego jest przekształcenie struktury dendrytycznej w strukturę globularną, co ma zasadniczy wpływ na własności mechaniczne wyrobu gotowego.

Zasadniczym osiągnięciem tej pracy jest wykorzystanie do pomiaru lepkości stali wiskozymetru, specjalnie zaprojektowanego do badań materiałów w wysokich temperaturach. Analiza reologii stopów żelaza w stanie stało-ciekłym wymaga zastosowania specjalnych wiskozymetrów wyposażonych w piece, których temperatura robocza zawiera się w przedziale od 1000 do 1500^{o}C. Badania przeprowadzono z wykorzystaniem wiskozymetru rotacyjnego z nieruchomym cylindrem zewnętrznym. Testy lepkości zostały poprzedzone doborem odpowiedniego kształtu tygla i wrzeciona oraz materiału, z którego zostały one wykonane, wytrzymującego temperatury ciekłej stali. Podstawowym kryterium doboru kształtu narzędzi było osiągnięcie prędkości odkształcenia postaciowego w zakresie do 20 s^{-1} w którym w trakcie pomiaru nie występuje zjawisko przepływu turbulentnego. Do pomiarów wykorzystano narzędzia wykonane z Al_2O_3. W pracy przedstawione zostały wyniki analizy reologicznej stali narzędziowej z gatunku 100Cr6 (PN ŁH15). Wyniki te obejmują zależności lepkości od prędkości odkształcenia postaciowego oraz temperatury. Biorąc pod uwagę sposób przeprowadzenia testów, polegający na równomiernym mieszaniu można założyć, że pomiar lepkości dotyczył stopu, w którym zaszła przemiana tiksotropowa.

Przeprowadzone badania pozwalające wyznaczyć własności reologiczne konieczne również do ustalenia wartości podstawowych parametrów technologicznych w procesach kształtowania tiksotropowego. W chwili obecnej prowadzi się szereg badań, których celem jest wdrożenie tej metody w przetwórstwie stopów żelaza. Formowanie tiksotropowe jest nowatorską metodą przetwórstwa stopów metali, posiadającą szereg zalet w porównaniu do klasycznych metod stosowanych w plastycznej przeróbce oraz odlewnictwie.

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1. Introduction

The research conducted allowed the rheological properties, which are necessary for the identification of the main technological parameter values of the metal-forming processes, to be determined [3]. Shaping in the semi-solid state, also known as the thixoforming processes, is an innovative method of processing metal alloys which has a great many advantages over the classical metal forming and foundry processes. Such processes are executed between the liquidus and solidus point of shaped metal alloys. The phase change which occurs during shaping and is connected with the solidification process results in the high sensitivity of material properties to changes of the technological parameters.

Nowadays, research is conducted with the practical application of this technology in steel processing as its goal [1,2].

Rheological analysis of ferrous alloys in the semi-solid state requires the use of a special viscometer with a furnace where the work temperature is within the range of 1000-1600°C. Measurements were carried out using a rotational viscometer with a stationary cup (outer cylinder). This paper presents the results of a rheological analysis of 100Cr6 (PN ŁH15) tool steel. The chemical composition of the steel is shown in Table 1, it was measured using an emissive – spark spectrometer.

TABLE 1
Chemical composition of 100Cr6 (PN ŁH15) steel

Fe	C	Si	Mn	P	S	Cr	Mo
96.5	0.978	0.229	0.320	0.006	0.008	1.52	0.0392
Ni	Al	Co	Cu	Nb	Ti	V	W
0.133	0.0093	0.0082	0.179	0.002	0.002	0.002	0.015
Pb	Sn	B	Ca	Zr	As	Bi	
0.025	0.0089	0.0010	0.0006	0.002	0.005	0.03	

Fig. 1 shows the liquid phase curve versus temperature in the solidification range.

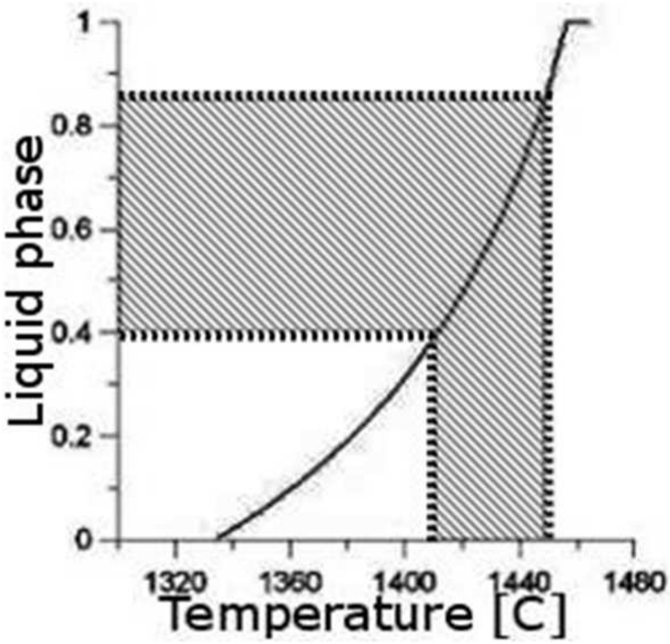

Fig. 1. Liquid phase in relation to temperature for 100Cr6 (PN ŁH15) steel

2. Viscosity measurement operating methods

The viscosity measurement of the alloys analyzed was performed using Searle's method [4,5]. In this method, the rod is set in motion and the cup is stationary. The cylinders are concentric, which means that both cylinders are showing 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 when measuring low-viscosity liquids at high rotational speeds.

The concentric cylinder methods proposed by Searle were used for this study. A diagram of the measuring system and a photograph of the actual are shown in Figures 2 and 3.

The geometrical parameters of the system are specified as follows: $R_e = 15$, $R_i = 7.5$, $L = 20$, $L' = 11$, $L'' = 11$ mm.

When measuring viscosity using the concentric cylinder method, the rheological parameters are defined as related to the radius of these cylinders.

Shear stress is defined as:

$$\tau(r) = \frac{M}{2\pi L r^2}, \tag{1}$$

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).

The representative shear stress is defined as:

$$\tau_{rep} = (\tau_i + \tau_e)/2, \tag{2}$$

where:
- τ_i – shear stress on the surface of inner cylinder ,
- τ_e – shear stress on the surface of outer cylinder.

Then:

$$\tau = \tau_{rep} = \frac{1+\delta_{cc}^2}{2\delta_{cc}^2}\frac{M}{2\pi L R_i^2 c_L} = C_{ss}M, \tag{3}$$

- c_L[-] - 'end – effect correction factor', which accounts for that part of the total torque occurring on the conical area of the apex of the rod (usually, we would use $c_L = 1.10$),
- C_{ss} [Pa/Nm; m^{-3}] – the conversion factor between M and τ, C_{ss} depends only on the geometrical dimension, i.e. R_i,
- δ_{cc} [-] – the ratio of the radii; the index 'cc' stands for 'concentric cylinder'.

The shear rate is defined by formula 4.

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

where:

- R_i[m] – the value of the inner radius,
- R_e [m] – the value of the outer radius
- ω [rad/s] – the rotational speed,
- r[m] – the distance from the rotational axis.

The representative shear rate:

$$\dot{\gamma}_{rep} = (\dot{\gamma}_i + \dot{\gamma}_e)/2, \tag{5}$$

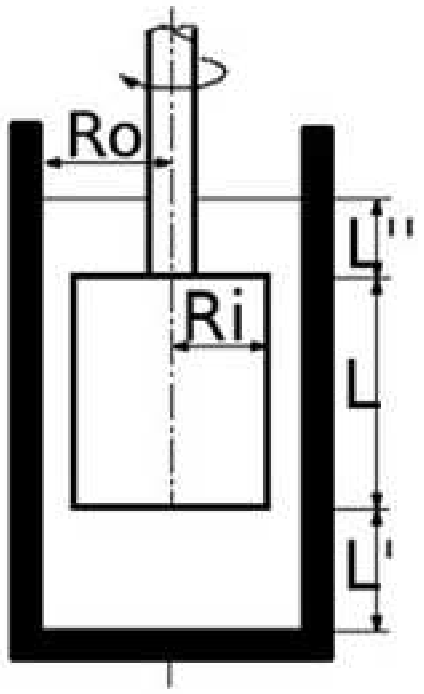

Fig. 2. Concentric cylinder measuring system [4,5,6]

Thus:

$$\dot{\gamma} = \dot{\gamma}_{rep} = \frac{\left(1 + \delta_{cc}^2\right)}{\delta_{cc}^2 - 1}\omega = C_{sr} \cdot n, \tag{6}$$

where

- δ_{cc} [-] – the ratio of the radii, i.e. the ratio of the radius of the outer cylinder to that of the inner cylinder,
- ω [rad/s] – the rotational speed,
- n [min^{-1}] – revolutions per minute,
- C_{sr} [min/s] – constant, as the conversion factor between n and $\dot{\gamma}$, C_{sr} depends only on the ratio of $R_i$to R_e.

For the geometry shown in Fig. 3, the values of C_{ss} and C_{sr}, calculated from formulas 3 and 6, are specified as follows: 630 Pa/mNm (assumed values of $c_L = 0.85$) and 0.314 min/s.

Fig. 3. The high-temperature rheometer FRS1600 used in this research

3. Modeling the viscosity of metal alloys in a semi-solid state

During the measurements taken using the rheometer described above, the correlation between shear stress and shear rate was determined. The nature of the results suggests the non-Newtonian behavior of the medium being studied.

In order to use the results of the measurements in a numerical simulation, it was decided to describe the flow curve with the help of a rheological formula based on the power model used e.g. in ProCAST software. The parameters of this formula were estimated for the temperature range and shear rate being studied. The model determines the non-linear dependency between viscosity η and shear rate $\dot{\gamma}$.

$$\eta = \eta_0 \cdot (K \cdot \dot{\gamma})^n, \quad \text{if} \quad \dot{\gamma} \geqslant \dot{\gamma}_0, \tag{7}$$

$$\eta = \eta_0 \cdot (K \cdot \dot{\gamma})^n, \quad \text{if} \quad \dot{\gamma} < \dot{\gamma}_0, \tag{8}$$

where:
- η_0 – viscosity if $n = 0$,
- γ_0 – the critical shear rate (the value of the shear rate; below this, there is no change to the value of the viscosity),
- K – the consistency of the material,
- n – shear rate sensitivity.

4. Investigation and analysis of the results

The viscosity measurements were conducted as temperature and shear rate functions. The viscosity of semi-solid metal alloys is sensitive to both these parameters. High sensitivity to temperature changes is the result of phase transition. Phase transition is the result of the solidification process, which strongly influences the shaping of the material. A strong dependence on the shear rate is the result of semi-solid properties. In pictures 4-7, the results of the measurements for 100Cr6 bearing steel were shown. Viscosity measurements were performed for a shear rate in the range of 0-10 s^{-1} and a temperature range from 1410 to 1450°C. In this range, (as in Figure 1) 100Cr6 steel has a liquid phase from 0.40 to 0.85.

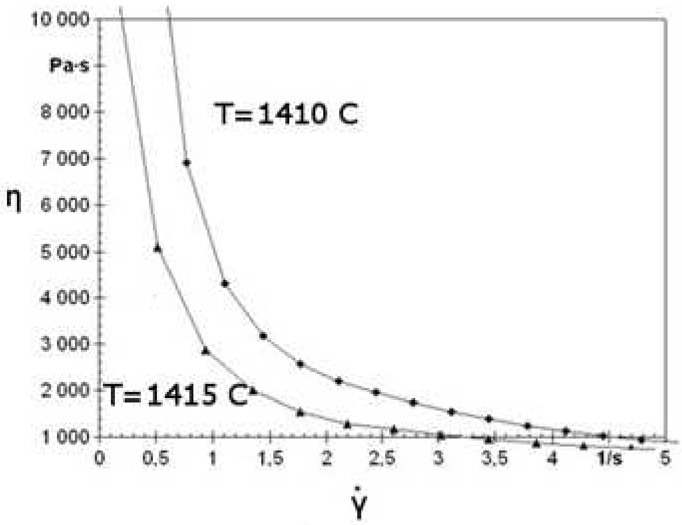

Fig. 4. Viscosity curves vs. shear rate for temperatures of 1410 and 1415°C

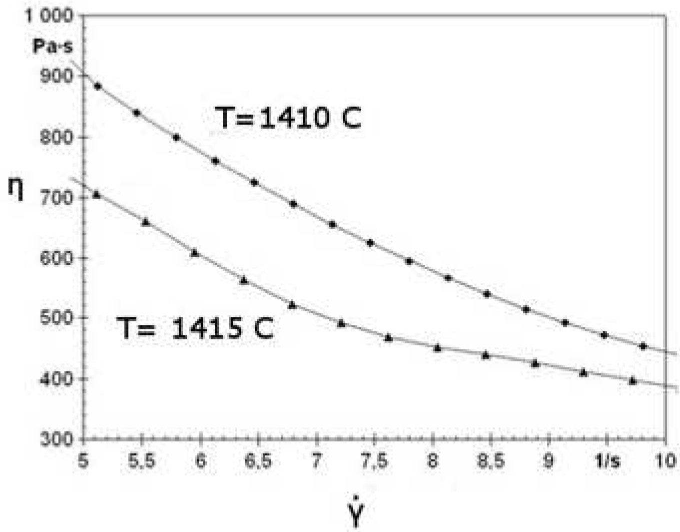

Fig. 5. Viscosity curves vs. shear rate for temperatures of 1410 and 1415°C

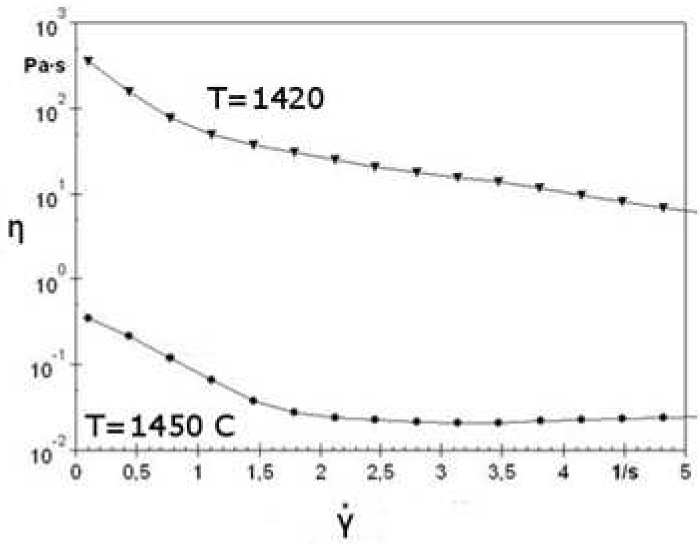

Fig. 6. Viscosity curves vs. shear rate for temperatures of 1420 and 1450°C

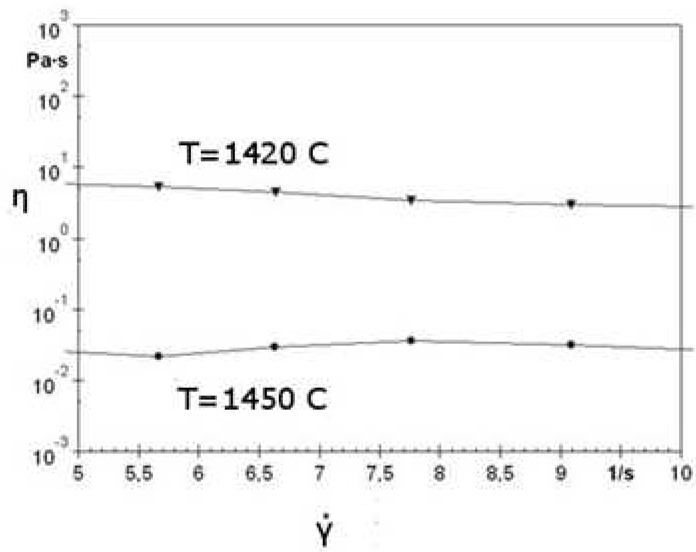

Fig. 7. Viscosity curves vs. shear rate for temperatures of 1420 and 1450°C

Figure 8 shows the comparison of viscosity curves obtained from the measurements and model (7-8) for bearing steel being studied. The curves are presented in a logarithm scale.

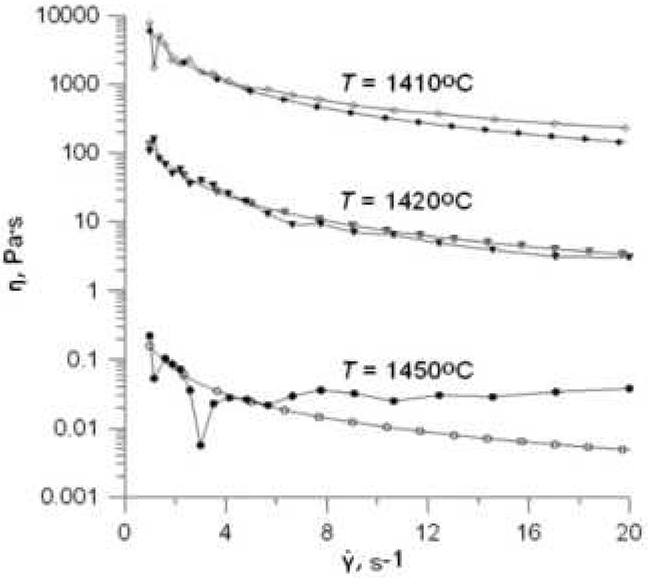

Fig. 8. Comparison of viscosity curves obtained from the measurements (dark points) and the model (7-8) (white points)

Table 2, presents the values of parameters μ_0, K and n, which were estimated for the power model (7-8). Approximation of these parameters using exponential function is shown in Figure 9.

TABLE 2

Metallic radii of rare earth metals and magnesium [12]

T[°C]	η_0[Pa·s]	K[s]	n[-]
1410	2180.14	0.42	-1.16
1420	393.98	3.63	-1.15
1450	4.92	71.84	-0.8

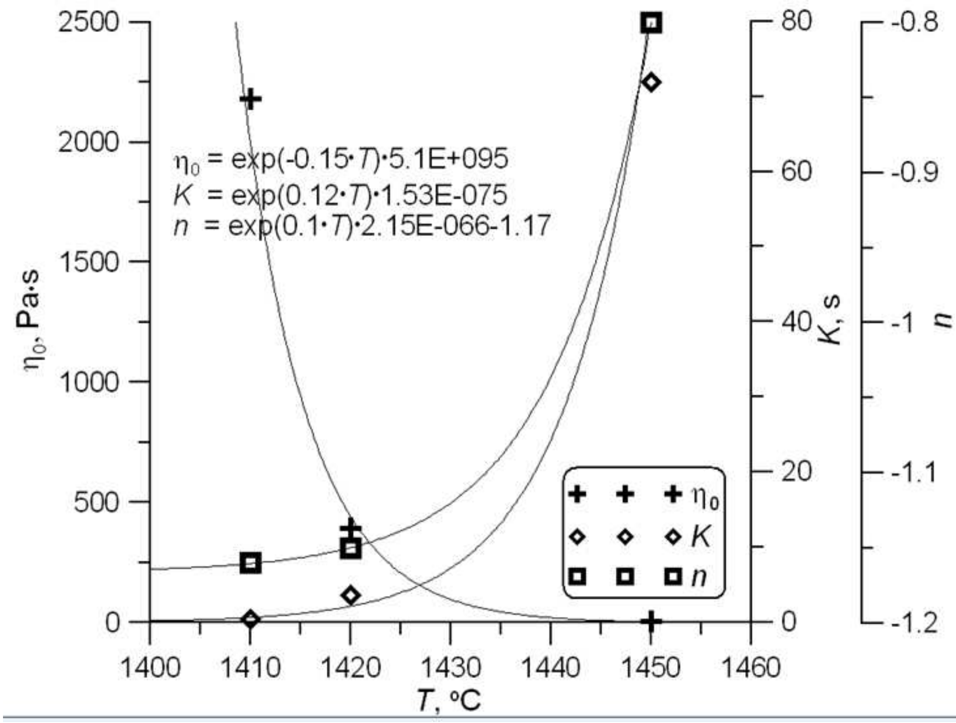

Fig. 9. Values of rheological parameters of 100Cr6 steel η0, K and n and their approximation using exponential functions

5. Conclusions

For this study, the measurements of the viscosity of selected ferrous alloys in a semi-solid state were conducted. The main result of the work is the application of a viscometer which was specially designed for material tests executed at extremely high temperatures in order to carry out the measurement of steel viscosity. Future investigation will be conducted with modified geometry for the rotating rod and the cup. Changing the geometry makes it possible to measure a higher range of shear rate avoiding slips on the surface between cylinders and medium.

A comprehensive analysis of rheological properties is crucial for the improvment of the numerical modeling of technological processes.

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