Characterization of solidus-liquidus range and semi-solid processing of advanced steels and aluminum alloy

Łukasz Rogal

Institute of Metallurgy and Materials Science of the Polish Academy of Sciences,
25 Reymonta St., 30-059 Krakow, Poland
e-mail: l.rogal@imim.pl

1. Introduction

Semi-Solid Metal processing (SSM) of steel is a method of producing near-net shape products [1]. This technology utilizes the thixotropic flow of metal suspension [2]. Metal alloys are suitable for thixoforming if they have globular microstructure in a wide solidus-liquidus range [3]. The thixo-route is a two-step process, which involves the preparation of a feedstock material of thixotropic characteristics and reheating the feedstock material to semisolid temperature to produce the SSM slurry, subsequently used for component shaping [4]. The goal of the applied thixoforming technology is to improve the properties of elements manufactured in one-step operation, ensuring their high structure integrity, superior to casting and similar to that of wrought parts. Nowadays, rheoforming and thixoforming of Al-based and Mg-based alloys are commonly applied in the industry [3-6]. In the case of high melting alloys, such as steels, this technology is in the implementation stage [4]. Several kinds of steels e.g. 100C6, X210CrW12, M2, C38, C45, 304 stainless steels have been investigated for the application for SSM processing [4-9, 11-13]. A globular microstructure is mainly obtained by Strain Induced and Melting Activation (SIMA), Recrystallization and Partial Melting (RAP) and the modification of chemical composition by grain refiners methods [4, 10-12].

2. Thixoforming procedure

The thixo-casts were produced using a specially build prototype device. A piston velocity of 1 m/s was applied. The locking force of the machine was 800 kN [14]. A billets (diameter – 30 mm, height – 30 mm) of 100Cr6 [12, 14], X210CrW12 [11] steels and 7075 aluminum alloys with additions of Sc and Zr [10] were placed in the coil of an inductive heating furnace at 1425 °C, 1250 °C and 632 °C respectively. The temperature of the feedstock was measured with S type thermocouple. The billet was then moved to a shot sleeve of a high-pressure die-casting machine and forced by a piston into pressing force 35 kN the die, made of M2 steel, pre-heated to about 180°C and sprayed with BN [14].

3. Characterization of 100Cr6 semi-solid range and microstructure after SSM

The 100Cr6 bearing steel used was produced by HSW Quality Steel Works. The steel was hot forged at approximately 1050°C. Next it was normalized at 880°C. The chemical composition was 0,9% C, 1,4% Cr, 0,4% Mn, 0,3% Si, 0,2% Cu (all in weight %). The curve in Figure 1 shows the dependence of the amount of liquid phase as a function of temperature for the 100Cr6 steel calculated from the DSC measurements. It shows melting start at 1307°C, and the end of melting process at 1497°C. Semi-solid processing was carried out at 25% of liquid phase which corresponded to the temperature of 1425°C [12].

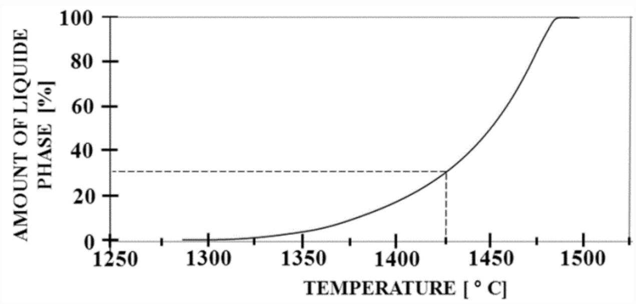

Fig 1 DSC heating curve for 100Cr6 steel after forging [12]

Thixoforming of 100Cr6 steel was carried out according to the procedure described in paragraph 2. Figure 2 shows an optical microstructure of a thixo-cast sample. The microstructure consisted of a primary globular grains (average size 343 µm) and secondary fine grains formed from a liquid phase during cooling (average size 20µm) [12].

Fig 2 Optical microstructure of 100Cr6 steel after thixoforming [15]

4. Characterization of feedstock and solidus-liquidus range of X210CrW12 tool steel

The X210CrW12 steel with a high content of chromium and carbon belongs to the ledeburite class of steels. Figure 3 shows an light microstructure in perpendicular direction to rolling.

It shows a primary carbide precipitation in the ferrite/pearlite matrix formed in bands parallel to the working direction. The average hardness of the billet is 220 HV_5. Such a steel was used as a feedstock for the thixoforming process [11].

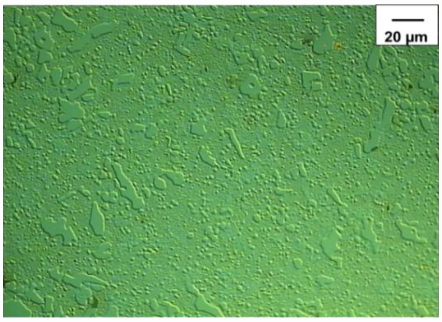

Fig 3 Microstructure of X210CrW12 steel after rolling – feedstock for thixoforming [15]

In Figure 4 the DSC curve (1) shows the endothermic effects, which occurred during heating at the rate of 20°C/ min. The curve (2) in Figure 4 shows the dependence of the calculated amount of liquid phase as a function of temperature. The melting point is determined as the onset of the endothermic peak.

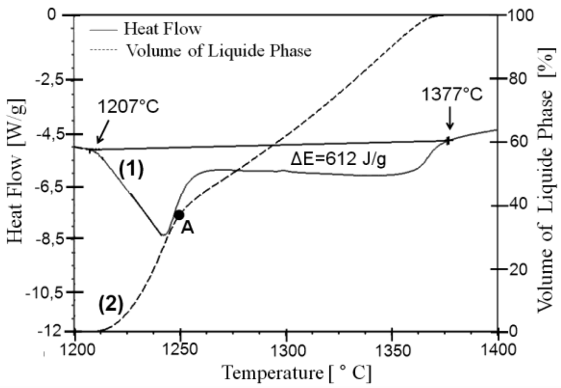

Fig 4 DSC heating flow and liquid fraction curves for X210CrW12 steel after rolling [11]

The start of melting is set at such a temperature when the heat curve falls away from the tangent line. The end of melting is set by the onset point on the other side of the peak. A strong endothermic effect begins at 1209°C and ends at 1379°C. The enthalpy of the melting process is 612 J/g. The analysis of the melting curve shows that in the range 1209°C - 1256°C (0-42% liquid phase) the enthalpy of melting is much larger than during the rest of the process. This effect is connected with melting of the eutectic. Over 1256°C mainly solid solution melts. This effect is also observable in curve (2), which shows different kinetics of changes of the amount of liquid phase as a function of temperature.

Semi-solid processing was carried out at 1250°C which corresponds to 38% of liquid phase in accordance with the DSC analysis (point A marked in Fig 4).

4.1. Microstructure of X210CrW12 after SSM

The optical microstructure of a X210CrW12 thixo-cast cross-section is shown in Figure 5. Visible austenite globular grains are surrounded by the eutectic mixture. The insert in Fig.5 shows a histogram of grain size. The distribution of grain size is within 15µm to 90µm. The largest fraction belongs to the grains in the range of 39 µm to 48 µm, indicating the average size of globules – 44 µm. The average hardness of the thixo-casts was 401 HV_5

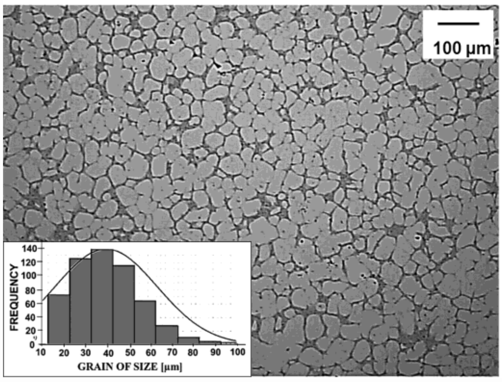

Fig 5 Microstructure of the thixo-casts made of steel X210CrW12 and quantitative analysis of grain size distribution [11]

5. Semi-solid range of Al7075 with Sc and Zr

The 7075 aluminium with 0.5 wt.% addition of combined Sc and Zr (7075ScZr) was cast in the form of rods of 30 mm diameter and 100 mm length into a copper die. The chemical composition of the alloy was determined to be: Mg- 2,83%, Cu-1,72%, Zn-5,86%, Zr, Sc – 0,5%, Al-88,73% (all in wt.%). The addition of 0.5 % of scandium and zirconium to Al-Mg-Cu-Zn alloys should cause the formation of $ZrAl_3$ and $ScAl_3$ phases. Crystallization nuclei based on Al_3 (Zr, Sc) phase play a significant role in refining of grains [15]. The curves in Figure 6 show the dependence of the amount of liquid phase as a function of temperature for the 7075ScZr calculated from the DSC measurements for heating. It shows that Al(α) melting started at 565°C for heating rate 15°C/min. The end of melting took place at 657°C for heating from the solid state. The semi-solid processing was carried out at 632°C, which corresponds to 25% of liquid phase in accordance with the DSC analysis (point A marked in Fig 6).

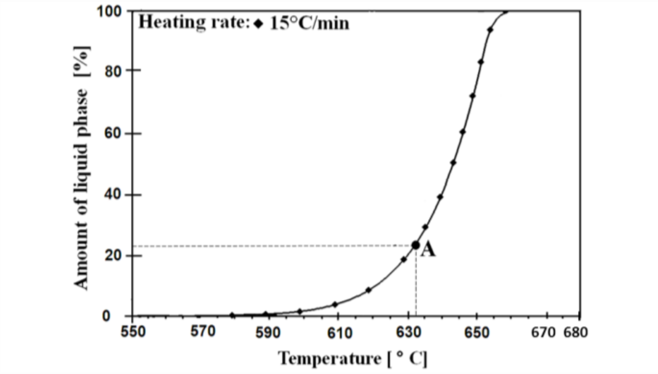

Fig 6 Liquid fraction vs. temperature curves for the 7075ScZr alloy obtained from heat flow vs temperature data recorded during heating from the solid state [10].

5.1. Analysis of thixo-formed 7075ScZr aluminium alloy

The optical microstructure of a thixo-formed part is shown in Figure 7. The microstructure was homogeneous, and consisted of globular grains surrounded by the eutectic mixture. The average size of α(Al) was 26 µm.

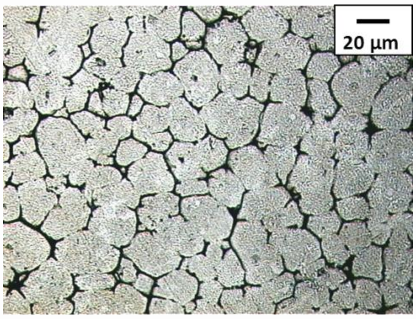

Fig 7 Microstructure of the thixo-casts made of steel 7075ScZr [15]

The precipitates of M ((Cu, Zn, Al)2Mg)) and T ((Cu, Zn, Al)49Mg32)) phases are present among the grains [10]. Amount of the eutectic mixture and precipitates of M and T phases was estimated from 28 to 32% [10].

6. Conclusions

Strain Induce and Melting Activated for steels as well as modification of molten 7075 aluminium alloy with 0.5% Sc and Zr appeared to be an effective methods of feedstock preparation for the thixoforming process. Suitable determination of solidus-liquidus range and technological parameters of process enable to obtain thixo-cast with defects.

7. References

[1] Rogal L, Dutkiewicz J, Czeppe T, Bonarski J, Olszowska-Sobieraj B., Characteristics of 100Cr6 bearing steel after thixoforming process performed with prototype device. China Transactions of Nonferrous Metals Society of China 2010; 20:1033-1037

[2] Spencer B, Mehrabian R, Flemings MC, Metall. Trans. 1972;3:1925-1932

[3] Czerwinski F, Magnesium Injection Molding, Springer 2008.

[4] Hirt G, Kopp R, Thixoforming Semi-Solid Metal Processing. Wiley-vch Publications (2009), ISBN 978-3-5273-2204-6

[5] Kopp R, Shimahara H, Schneider JM, Kurapov D, Telle R, Munstermann S, Lugscheider E, Bobzin K, Maes M, Characterization of steel thixoforming tool materials by high temperature compression tests. Steel Research International 2004;75: 569-576

[6] Cezard P, Sourmail T,Thixoforming of Steel a State of the Art from Industrial Point of View. Solid State Phenomena 2008;141: 25-35

[7] Atkinson H, Rassili A, Thixoforming Steel. Shaker Verlag Publications (2010), ISBN 978-3-8322-9133-4

[8] Puttgen W, Hallstedt B, Bleck W, Loffler JF, Uggowitzer PJ, On the microstructure and properties of 100Cr6 steel processed in the semi-solid state. Acta Materialia 2007; 55: 6553-6560.

[9] Rogal Ł., Dutkiewicz J., Globular microstructure formation in X210CrW12 steel for semi-solid processing using plastic deformation and boron modification, Archives of Metallurgy and Materials, 2012; 58: 765-769.

[10] Rogal Ł., Dutkiewicz J., Atkinson H.V., Lityńska-Dobrzyńska L., Czeppe T., Modigell M., 2013, Characterization of semi-solid processing of aluminum alloy 7075 with Sc and Zr additions, Material Science and Engineering A, 580: 362-373

[11] Rogal Ł., Dutkiewicz J., 2012, Heat treatment of thixo-formed hypereutectic X210CrW12 tool steel, Metallurgical and Materials Transactions A 43A: 5009-5018

[12] Rogal Ł., Dutkiewicz J., 2012, Effect of annealing on microstructure, phase composition and mechanical properties of thixo-cast 100Cr6 steel, Mat. Characterization 68:123-130

[13] Rogal Ł., Dutkiewicz J., 2011, Transmission electron microscopy studies of X210CrW12 and 100Cr6 thixo-cast steels, Solid State Phenomena 186: 311-314

[14] Rogal Ł., Dutkiewicz J., Czeppe T., Bonarski J., Olszowska-Sobieraj B., 2010, Characteristics of 100Cr6 Bearing Steel after Thixoforming Process Performed with Prototype Device, Transactions of Nonferrous Metals Society of China 20: 1033-1036

[15] Rogal Ł., 2012, Developing steel and aluminum alloy thixoforming technology and characterization of the obtained thixo-casts, PhD thesis 10-140