17th International Conference
of Polish, Czech and Slovak Foundrymen
13 - 16.04.2011 r.
Książ Castle

DSC and TA characteristics of the inoculated Al-Zn20 alloy

W.K. Krajewski [a,*], J. Buraś [a], G. Piwowarski [a], K. Haberl [b], D. Tsivoulas [c]
[a] Faculty of Foundry Engineering, AGH University of Science and Technology, Reymonta 23, 30-059 Krakow, Poland
[b] Department of Metallurgy, University of Leoben, Franz-Joseph-Strasse 18, A8700 Leoben, Austria
[c] School of Materials, The University of Manchester, Grosvenor Street, M1 7HS, Manchester, UK
*Corresponding author. E-mail address: krajwit@agh.edu.pl

Abstract

The paper brings information about inoculation process of the sand-cast binary Al-20 wt% Zn (AlZn20) alloy, inoculated with Ti-containing grain-refiners, i.e. AlTi5B1, AlTi3C0.15 and (AlZn)-Ti3 master alloys (MA). During the experiments thermal analyses (TA) and differential scanning calorimetry (DSC) were used. Macrostructure observations were performed using light microscopy (LM). On the basis of the performed examinations it was found that all the MAs used in this work caused significant grain refinement, which initiates via a heterogeneous nucleation mode. The obtained structure fineness of the inoculated AlZn20 alloy should positively influence its ductility, which is of great importance for the practical application of this group of foundry alloys.

Keywords: Theory of Crystallization, Solidification Process, Thermal Analysis, Differential Scanning Calorimetry, Al-Zn Cast Alloys

1. Introduction

Inoculation is a commonly used melt treatment before casting into a mould, which allows obtaining a higher population of grains, Fig. 1, which positively influences the ductility of the alloys. The aim of the master alloy additions in the Al melt prior to solidification is not confined simply to refining the grain structure. The main goal is to obtain a uniform and equiaxed microstructure free from coarse, columnar grains which are detrimental to the final properties of the material, Fig. 2. There have been several studies on the Al-Zn20 system in the past [1-3]. The present work continues the research on the optimisation of master alloy additions in the Al20Zn base alloy, towards an environmentally friendly solution that entails energy requirement minimisation and weight reduction. DSC measurements hold a major role in the examination of these materials and this is why it will be emphasised in this paper studied here, focusing on various master alloy additions. The paper brings also cooling curves from Thermal Analyses (TA) obtained during the solidification process in sand moulds of the non-inoculated and inoculated Al-Zn20 alloy.

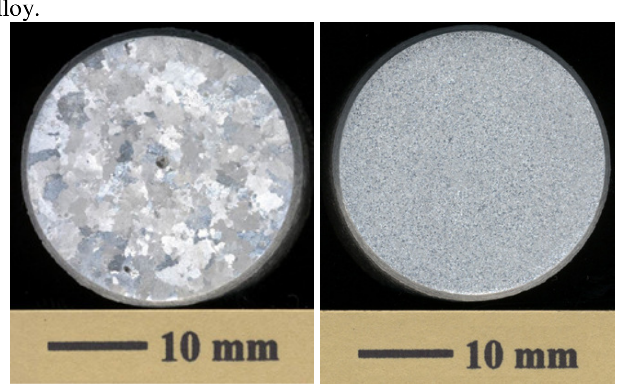


Fig. 1. Light microscopy picture (LM) of the AlZn20 sand-cast alloy. Left – initial, non-inoculated; visible coarse-grained structure. Right – inoculated with TiBAl MA (0.04 wt% Ti); visible highly refined macrostructure [9]

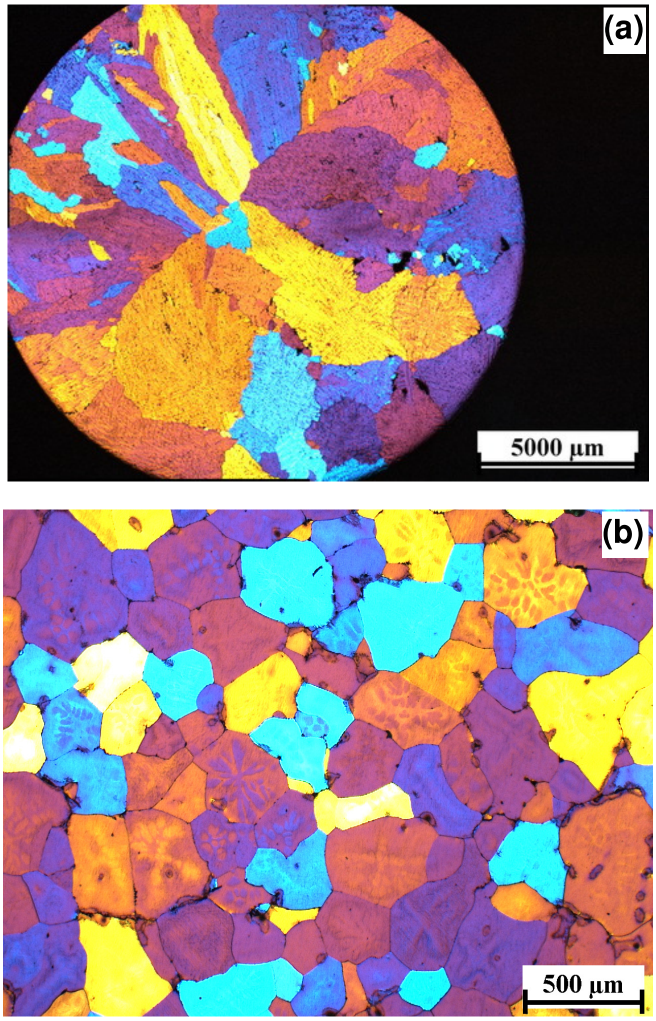


Fig. 2. LM picture (Zeiss Axio Imager A.1m) of the AlZn20 sand cast alloy, Barker's etchant. (a) – initial alloy, non-inoculated; visible coarse-grained structure; (b) the AlZn20 alloy inoculated with 400 ppm Ti, introduced to the melt with the AlZn-Ti3 MA [3, 4].

2. Materials and methodology

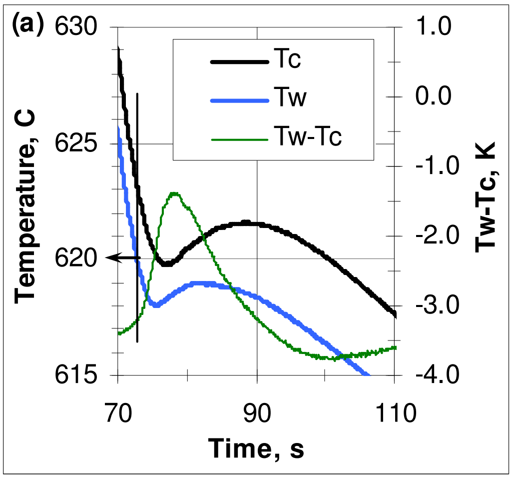
The base alloy Al-20wt%Zn was prepared from electrolytic aluminium (minimum purity 99.96%) and electrolytic zinc (99.995%). The melting was performed in an electric resistance furnace, in a clay-graphite crucible of 1.5 litre capacity. The Al-Zn20 melt was superheated to ~740^{o}C and then the melt was degassed by bubbling Ar through it for 10 min. After removing the dross from the surface of the melt, a master alloy was added and the melt was held at the same temperature for 2 min. Then the melt was stirred for the next 2 min with an alumina rod and finally, the alloy was cast into a dried sand mould to obtain cylindrical tensile test-pieces (working part ∅12x60 mm). The base Al-20wt%Zn alloy was inoculated with four different grain refiners in order to determine their effects on the DSC curves during a full thermal cycle. These additions were made from the following master alloys (MAs); Al-3wt%Ti-0.15wt%C (TiCAl), Al-5wt%Ti-1wt%B (TiBAl), Zn-4.6wt%Ti (ZnTi4) and Al-20wt%Zn-3wt%Ti (AlZn)-Ti3. The first two MAs were commercially available, while the last two were prepared in the laboratory as described elsewhere [1, 6]. The amount of Ti present in the final alloy was in the range of 500 ppm. More details can be found in a previous report [3]. DSC analysis was carried out with SDT Q600 TA device, under an Ar atmosphere to avoid oxidation of the samples. Samples were heated up between 700-800 ^{o}C at a rate of 10 K/min and cooled down in the same manner. The holding time at the higher temperature was 5 min, to allow for some time to equilibrate the temperature between the sample and reference pans before the start of the cooling stage. An alumina crucible was used to hold the sample, while the reference pan was left empty. Samples were cut from the as-cast tensile test specimens, with dimensions ∅4x1.5-2 mm (~70-80mg).

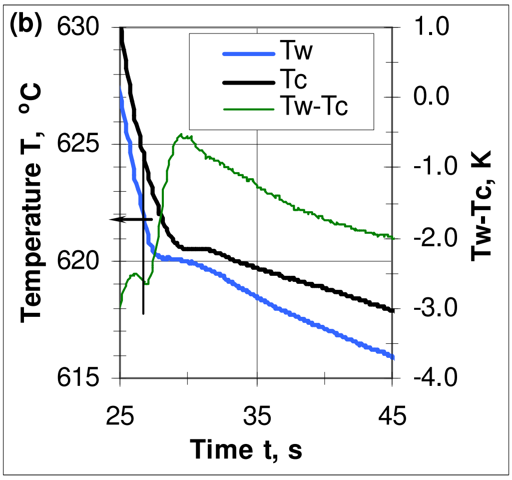
Data analysis was performed using the built-in TA software of the DSC instrument which was used to calculate the peak onset and offset points, as well as the area of the peaks. Several measurements were averaged to obtain the final values of each alloy.

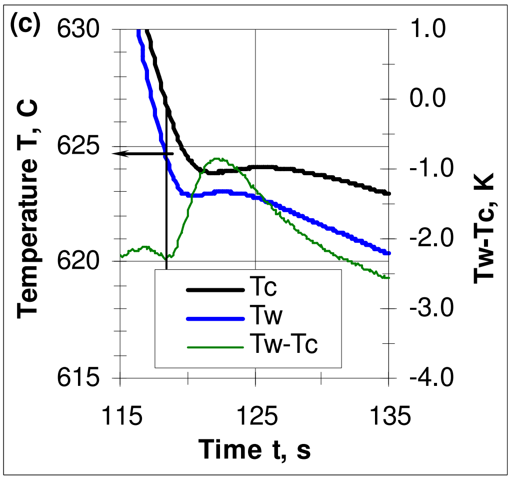
To monitor the solidification process and to register cooling curves of the solidifying AlZn20 samples of dimensions ∅32 x 80 mm, 2 thermocouples NiCr-NiAl0.5 ∅0.20 mm were mounted within the mould. i.e. Tc – registering temperatures in centre of the solidifying sample and Tw – registering temperatures within the sample but near the mould wall, positioned on the same height as the Tc thermocouple. Temperatures were recorded using a multi-channel recorder Agilent 34970A (Agilent Technologies Inc., USA) with an accuracy of ± 1^{o}C. Specimens for LM examinations were ground on abrasive paper (grit 200-1000) and then were polished using sub-microscopic aluminium oxide in water-alcohol suspension. The AlZn20 samples, used in macrostructure examinations, were etched with Keller's or Barker's reagent. LM observations of microstructures were performed using Leica-DM IRM and Zeiss Axio Imager A.1m microscopes.

3. Results

The cooling curves of the initial, non-inoculated AlZn20 alloy and its inoculated alternatives are presented in Fig. 3 (a) – (d). It can be seen from Fig. 3 that all the used master alloys cause increase of the nucleation temperature Tn (pointed out by arrows), i.e. from about 620 ^{o}C for the initial alloy to about 625 – 626 ^{o}C for the AlZn20 alloy inoculated with the TiCAl and TiBAl master alloys, and to about 622 ^{o}C when using the AlZn-Ti3 master alloy. At the same time one can observe a decrease in the undercooling, or even the lack of it. However, differences between all the master alloys on the measured cooling curves are clearly seen. From this it appears, that the AlZn-Ti3 master alloy has rather moderate influence in comparison with the TiBAl and TiCAl master alloys, though it causes significant grain refinement. On the other hand, it can be concluded, that the grain refinement process should be performed not to achieve the grain refinement *per se*, but to improve ductility and enhance other properties which are of interest to the end users, e.g. tensile strength or damping properties [8, 9]. The above-mentioned tendency of the nucleation start temperature to increase is also confirmed by the DSC examinations. Namely, from Fig. 4 (b) it can be seen that the initial AlZn20 alloy has the lowest Tn temperature (black colour curve). On the other hand, all the other master alloys caused a comparable increase of the Tn value, although the TA measurements gave slightly different results, as described earlier.







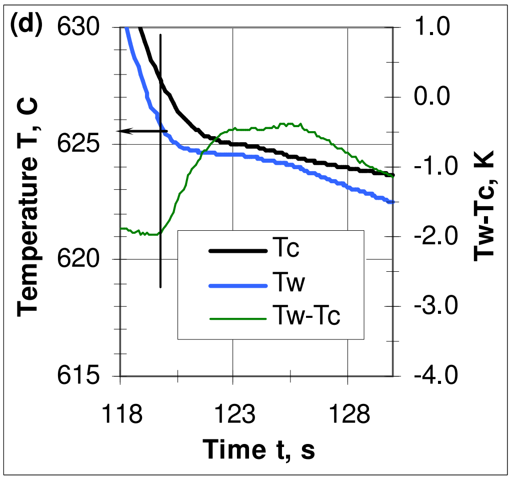


Fig. 3. Cooling curves of the sand-cast AlZn20 alloy. (a) initial, non-inoculated alloy, nucleation start temperature Tn ~ 620 °C; (b) AlZn20 + AlZn-Ti3 MA, Tn ~ 622.0 °C; (c) AlZn20 + TiCAl MA, Tn ~ 624.8 °C; (d) AlZn20 + TiBAl MA, Tn ~ 625.5 °C;

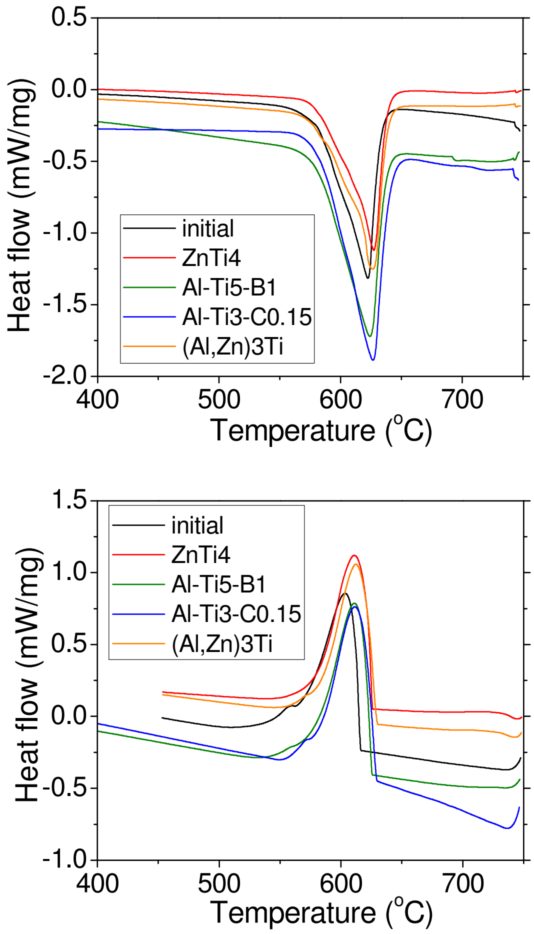


Fig. 4. Transformation peaks for all the alloys during: (a) melting and (b) crystallization.

4. Conclusions

Based on the examinations of the TiBAl, TiCAl and AlZn-Ti3 master alloys' performance as grain refiners in the sand-cast AlZn20 alloy, it can be concluded that all the master alloys cause significant grain refinement. As it appears from Fig. 3 they increase the nucleation start temperature Tn, decreasing the undercooling at the same time. Additionally, a decrease of temperature recalescention on the registered cooling curves is clearly visible. According to these findings, it can be concluded that nucleation of solidification in the AlZn20 alloy inoculated with the TiBAl, TiCAl and AlZn-Ti3 MAs has a quite heterogeneous nature.

The results obtained during thermal analysis were also supported by the DSC examinations. However differences were reported between values of the Tn temperatures obtained in TA and DSC examinations. As it was discussed in detail in [5] and [6], the possible reason is the sample mass used during the DSC experiments. Elucidation of the mentioned differences requires undertaking more detailed DSC examinations in the future.

Acknowledgements

The authors acknowledge The European Community for financial support under Marie Curie Transfer of Knowledge grant No. MTKD-CT-2006-042468. The provision of laboratory facilities in the Faculty of Foundry Engineering – AGH UST Krakow and Institute of Metallurgy and Materials Science – PAN Krakow is also kindly acknowledged.

References

[1] W.K. Krajewski, A.L. Greer, J. Zych, J. Buraś, Effectiveness of Zn-Ti based refiner of Al and Zn foundry alloys Foundry Trade Journal, vol. 180, No. 3642, (2007) 97-100.

[2] K. Haberl, W.K. Krajewski, P. Schumacher, Microstructural Features of the Grain-refined Sand Cast Alloy AlZn20, Archives of Metallurgy and Materials, vol. 55, No. 3, (2010) 837-841

[3] K. Haberl, Examination of the influence of various grain refiners on an AlZn20-alloy, Marie Curie Host Fellowship report, Krakow, June 2009, pp. 13 (unpublished).

[4] K. Haberl, Examination of AlZn20 alloyed with various grain refiners, Marie Curie Host Fellowship report, Leoben, May 2010, pp. 9 (unpublished).

[5] D. Tsivoulas, Development of Al-Zn and Al-Mg cast alloys and composites Marie Curie Host Fellowship report, Krakow, June 2010, pp. 21 (unpublished).

[6] D. Tsivoulas, T. Czeppe, W.K. Krajewski, DSC examinations of the Al – 20 wt% Zn sand-cast alloy inoculated with Ti-containing grain refiners. Inżynieria Materiałowa, vol. 175, Nr 3, (2010) 590-593

[7] W.K. Krajewski, Structure and properties of high-aluminium zinc alloys inoculated with Ti addition (retrospective coverage), Archives of Foundry, vol. 5, No. 15 (2005) 231–240

[8] W.K. Krajewski, J. Buras, K. Haberl, P. Schumacher, Development of environmentally friendly cast alloys and composites. High-Zinc Al based cast alloys. Archives of Foundry Engineering, vol. 10, special issue 1 (2010) 431-434

[9] J. Buraś, The influence of grain refinement on damping properties of selected aluminium zinc cast alloys. PhD thesis, AGH University of Science and Technology, Krakow 2010 (in Polish)

Charakterystyki DSC i TA zmodyfikowanego stopu AlZn20

Streszczenie

Praca dotyczy procesu zarodkowania stopu AlZn20, odlewanego do formy piaskowej, przy pomocy zapraw zawierajacych Ti AlTi5B1, AlTi3C0.15 i (AlZn)-Ti3. W czasie eksperymentów stosowano analizę termiczną (TA), różniczkową kalorymetrię skaningowa (DSC) oraz obserwacje struktur przy pomocy mikroskopii świetlnej (LM). Na podstawie wyników badań stwierdzono, że wszystkie zaprawy powodują rozdrobnienie struktury wskutek heterogenicznego zarodkowania. Uzyskane rozdrobnienie struktury powinno pozytywnie wpływać na plastyczność zmodyfikowanego stopu, co ma duże znaczenie dla praktycznego zastosowania tej grupy stopów odlewniczych.