International Journal of Engineering & Technology, 8 (1.1) (2019) 31-35



## **International Journal of Engineering & Technology**

Website: www.sciencepubco.com/index.php/IJET



Research paper

# The DTA Curves for Melting of Hypereutectic AlSi Alloy

M. S. Wahab<sup>1\*</sup>, Aqeel Ahmed<sup>2</sup>, A. A. Raus<sup>3</sup>, K. Kamarudin<sup>4</sup>, E. Z. Radzi<sup>5</sup>

<sup>1</sup> Faculty of Mechanical and Manufacturing Engineering, Universiti Tun Hussein Onn Malaysia, Parit Raja, 86400, Malaysia. \*Corresponding author E-mail: saidin@uthm.edu.my

#### Abstract

Hypereutectic Al-Si alloy having >12 wt% of Si, because of coarse and primary angular Si particles, it is very difficult to improve its mechanical, thermal properties and any phase change. Thermo Mechanical properties and phase change of material is completely depend on melting & solidification of material. Differential thermal analysis is a relatively profound method for studying solidification and melting of alloys. It provides an exact estimation of the characteristic temperature of any phase changes with latent heat release or temperature variation. Such method is used here to a study the melting behavior of the Al-20Si and Al-25Si alloys with respect to temperature variation. Thermo gravimetric Analysis (TGA) resulted, the entire changes in mass of 1.57 mg and 1.43 mg for AlSi20 and AlSi25 alloys respectively. Differential Thermal Analysis (DTA) graph resulted the melting temperature of Al-20Si is 661 °C and for Al-25Si is 680 °C. These values are differing from the value of actual melting temperature of Al-Si alloy. The selection of heating rate is important to achieve better melting behavior results.

Keywords: Differential Thermal Analysis; Hypereutectic Al-Si Alloy; Melting Temperature and Thermo gravimetric Analysi.

#### 1. Introduction

Hypereutectic AlSi alloys hold > 12.6 wt% of Si. The regular example of such alloy employed in industry are 4928A (17-19% Si) and 4928B (23-26% Si). The higher percentages of silicon correspondingly decrease thermal expansion and increase thermal conductivity [1-2]. However; not as regularly employed as hypoeutectic alloys, they possess many number of properties that mark them good-fitted in many specific applications. They are generally described as having excellent wear resistance, weight reduction and thermal conductivity in comparison with hypoeutectic alloys. Hypereutectic Al-Si alloys are one of the extensively utilized alloys in pistons, cylinder heads, cylinder liners and cylinders of the automobile engines. It is a direct result of numerous great properties, for example: high specific strength, low coefficient of thermal expansion, low density, excellent wear resistance, corrosion resistance, and many more. Whereas in contrast with hypoeutectic Al-Si alloys, it possesses ductile primary Al which makes and promulgates dendritically, and a needle-like brittle hard eutectic Si phase.

The principal family of Al–Si foundry alloys have Si from 5 to 25 wt.%, with significant components: Cu, Ni and Mg additions. Discussing the Si presence in weight percent (wt.%), the Al-Si alloys categorized into three major groups: hypoeutectic having <12% Si, eutectic from 12-13% Si and hypereutectic ranging from 14-25% Si. [3-4]. The AlSi eutectic system take place at a composition of  $\sim 12$  wt% of Si at temperature  $\sim 633^{\circ}$ C ( $\sim 1171^{\circ}$ F) (Figure 1). The expression "hyper" usually means alloys having more than  $\sim 12\%$  silicon. As will be clarified thusly, a more fitted significance of the word hypereutectic in that background might be alloys that have primary silicon crystals. Particular Al-Si alloy microstructures are shown in Figure 1, showing the hypoeutectic (b), eutectic (c) and hypereutectic (d) silicon phase morphologies.

The presence of coarse and primary Si particles in the microstructure of the AlSi hypereutectic alloys have been identified as the main limitation for their industrial use. Indeed, even with the utilization of silicon modifiers and high cooling rates, the primary Si particles can only be diminished in size. But adding some other alloying elements in the hypereutectic AlSi alloys and forming such alloy base composites can improve mechanical properties and sidestep these limitations. The alloying components Cu, Mg, Fe, Ni, Mn, and so forth can be used to build the distinctive properties of hypereutectic AlSi composites. Copper has impact to influence the hardness and strength of hypereutectic AlSi alloy at either heat treated or non-heat treated and also at ambient or at elevated temperature. It also increases the hardness and cause to improvement in the machinability of the hypereutectic AlSi alloy. Mg in the hypereutectic AlSi alloy resulted significant strength and also increases the work hardening. Mg also imparts good corrosion resistance, gives very high strength and is a source for providing good weldability. The expansion of Si and Mg together in the hypereutectic AlSi alloy enhances the weldability, however; in some AlSi alloys, just because of good thermo-mechanical properties, the maximum addition of Mg can be 4-8 wt%. Nickel cause to enhance the hardness while zinc expansion has no any impacts, it neither increment nor diminish the mechanical properties of hypereutectic AlSi alloy. The addition of Manganese cause to improve the tensile strength. Also significance enhancement in the low cycle fatigue resistance and increase corrosion resistance of the hypereutectic AlSi alloy. However; until now Fe has not been counted as an alloying component. [5-6].

As per [6], regarding the thermal analysis for influence of alloying elements on the Al–Si cast alloys, Mg, Cu, and Si are significantly articulated and must be considered during the practical implication of thermal analysis. By expanding Si percentage, a decrement in the depression values detected whereas the microstructure remains relatively unaffected. Increasing Cu content prompts to upper depression



values and to a kind of over modified eutectic microstructure. Magnesium spectacles the most perilous performance with respect to modification control by means of thermal analysis. Because, the eutectic depression rely very much on the Mg content and can't be related with the subsequent microstructure. But the collective result of Cu and Fe concluded that both components are not dependent on each other and can be recognized individually. The temperature for precipitation of the AlSi eutectic changes significantly, 575 °C for Ba¨ckerud et al. [7] and 562 °C for Samuel et al. [8] in contrast with the point that this temperature is depressed by increasing the minimal Si percentage of the alloy [9].

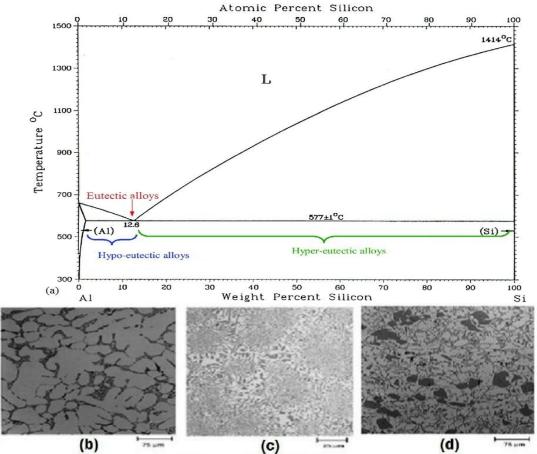


Fig. 1: AlSi Alloy Phase diagram (a), Typical Microstructure of Hypoeutectic (b), Eutectic (c) and Hypereutectic (d) AlSi Alloy

Thermal analysis is employed to found thermodynamic properties which are important for understanding the performance of material under different heating & cooling rates and under inert, reduction or oxidation atmosphere or under different gas pressures. Thermal analysis encompasses a group of techniques where the properties of material are studied as they change with temperature. To examine the thermo-physical properties numerous approaches are generally used: differential thermal analysis (DTA), differential scanning calorimetry (DSC), thermo gravimetric analysis (TGA). In DTA and TGA, the specimen and the reference material are heated in one furnace. The temperature difference of both materials is recorded during programmed heating and cooling cycles. The critical distinction amongst DSC and DTA gear is that the last is regularly utilized for the subjective estimations and it is stronger due to sensitive materials utilized for sample holders, heat conduction path and so on. The sample holder in the DTA apparatus is much inexpensive than the sample holder in the DSC apparatus and is acclaimed for the examination of materials with an unknown relation to contamination between the crucible and the sample holders [10]. Thermal analysis is a powerful technique that has been effectively used to examine and find characteristic temperatures, fraction solids and latent heats during the solidification process. Thermal analysis has been practically and effectively used for Al–Si hypocutectic alloys; however, the Al–Si hypereutectic alloys have not been as investigated.

Due to the above-mentioned opinions, it is of vital importance to conduct research to know the melting behavior as well as liquid state characteristics of the Al–Si hypereutectic alloys. This will enable the development of novel innovations to completely refine the essential Si morphology, which thus will expand the interest for the Al–Si hypereutectic composites. The present research investigation was conducted to understand in more detail about the thermal characteristics of the Al–Si hypereutectic alloys.

#### 2. Materials and Experimental Details

DTA and TGA for AlSi20 and AlSi25 alloys were performed by using Thermo gravimetric Analyzer apparatus LINSEIS L81 / 1550. This apparatus provides the evolution of temperature difference  $\Delta T$  between the sample and reference thermocouples as function of the sample thermocouple temperature.  $\Delta T$  is set in  $\mu V$  signal for DTA and in mg for TGA with temperature. Experiments consisted in heating to a maximum temperature about 1150 °C for AlSi20 alloy and AlSi25 alloy above the nominal liquids of them and holding at that temperature for 300 s followed by cooling to room temperature.

Both hypereutectic AlSi alloys were supplied from Chengdu Best New Materials Co.,Ltd China with details mentioned in below Table 1. To record DTA and TGA results, the material was used as received from the supplier.

Care was taken to prepare samples of about the same weight so that they should have the same weight and give fully comparable DTA records.

Table 1: Al-Si Allovs details

Item	Particle Size	Chemical Composition
Aluminum -Silicon Alloy (Si: 20%)	6~8 um	Al:78.665
		Si:20.835
		Zn:0.034
		Fe:0.215
		Other:0.033
Aluminum -Silicon Alloy (Si: 25%)	6~8 um	Al:74.680
		Si:24.835
		Zn:0.033
		Fe:0.215
		Other:0.033

#### 3. Results and Discussion

This study reports the melting behavior of two Al-Si alloys having Si contents at about 20 and 25 wt% by using DTA and TGA techniques.

#### 3.1 AlSi 20

Figure 2 shows the TGA and DTA and their derivatives curves of the AlSi20 alloy. The heating rate for the TGA & DTA experiments was also  $10\,^{0}$ C / min, while the maximum temperature was  $1050\,^{\circ}$ C for heating the sample. The mass for the sample weighted 58.4 mg. The graph for TGA recorded the changes in mass (gain / loss) and for DTA in  $\mu$ V with respect to temperature. As per the TGA graph, there isn't any considerable change is recorded in the starting, however; at temperature  $539.5\,^{\circ}$ C, an increment is observed in the graph line and continues up to  $681.7\,^{\circ}$ C. The gain in mass is  $1.57\,^{\circ}$ mg during this whole reaction.

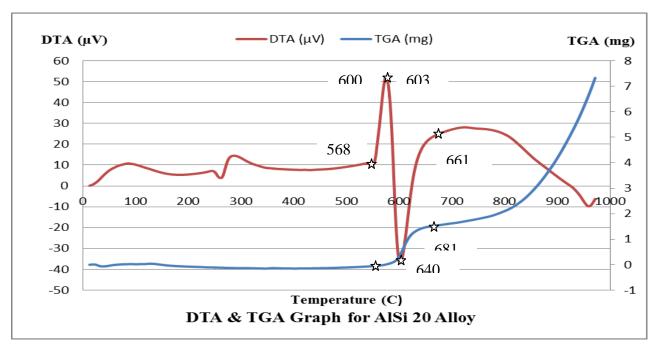


Fig. 2: DTA & TGA with their derivatives for AlSi20

While as per the DTA graph, the 1st reaction (exothermic) starts at around 568  $^{\circ}$ C and ends at around 600  $^{\circ}$ C. In this reaction the crystal structure changed in between these two temperature readings and considered as sharp crystallization phase transition. The 2nd exponential line defines the endothermic reaction which starts at 603  $^{\circ}$ C and ends at 640  $^{\circ}$ C.

From resulted graph it is considered as another sharp transition but from crystallization to melting (liquid) phase. This sharp transition progresses to another exothermic reaction and finishes at 661 °C where AlSi20 alloy completely melt. The last reaction is considered as the melting phase and the temperature 661 °C is considered as the melting temperature of AlSi20 alloy. As per [5], the Si content has an obvious influence on the thermal behavior of the AlSi alloys. But value achieved in this case is almost near to the value of actual melting temperature of the AlSi alloy. The obtained value is higher than the actual value of the AlSi alloys and is because of selection of heating rate as 10 °C / min. Any variation in the recorded value and actual value in the melting temperature is because of the heating rate [11]. The heating rate for obtaining DTA and TGA is very important to obtain accurate values in the thermal analysis of the materials.

#### 3.2 AlSi 25

Figure 3 shows DTA, TGA and with their derivatives curves for the AlSi25 alloy. The Al-Si-based alloys and Al have melting points of about 580 °C and 660 °C respectively, and large latent heats of about 400 J/g [12]. The heating rate for the graph was 10 0C/min,

while the maximum temperature was 11500 °C for heating the sample. The mass for the examining the sample weighted 60.2 mg. The recorded TGA graph shows the changes in mass (gain / loss) with respect to temperature. In the beginning of the graph; the weight of the sample is considered as 0 mg to record the changes in the mass of the sample.

To start with first development, there is consistently decrement in the TGA graph and at about 550 °C; the loss of -0.44 mg is recorded in the mass of the sample. But at around 580 °C; an increment in the mass is observed and it continues upto 680 °C and change the mass of the sample material about 1.0 mg.

At about 680 °C, the total gain in mass is 1.03 mg, however; in between these two temperatures, the total change in mass is 1.43 mg. Discuss on the exothermic and endothermic reactions (DTA curve), the 1st reaction (exothermic) starts at around 150 °C and ends at around 250 °C. This is the crystallization phase of the alloy and during this reaction, the crystal structure changes from one phase to another phase. In other words it is considered as phase transition of the alloy which cause change in the weight and can be noticed in the TGA graph. The 2nd exponential line defines the endothermic reaction; it starts at 580 °C and ends at 640 °C. During this, a very sharp exponential line is recorded and called as sharp phase transition which continues to another reaction and ends at 680 °C. This completely changes the crystalline phase of the material to melting phase.

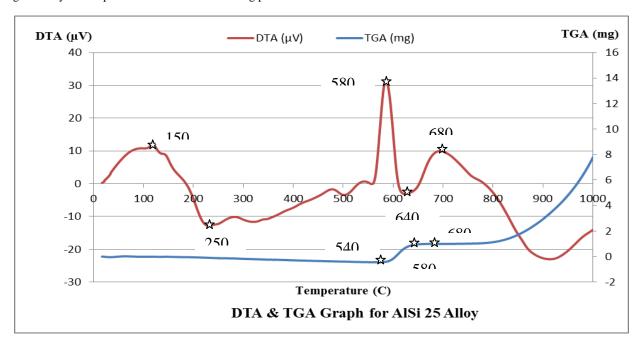


Fig. 3: DTA & TGA with their derivatives for AlSi25

#### 4. Conclusion

DTA and TGA are widely used for measurement of alloy solidification and melting behavior. A very simple model developed earlier [13-14] has been used to estimate solidification kinetics from the DTA curves. In that model, the temperatures of the sample of the cup and of the thermocouple are assumed identical for the sample holder on the one hand and the reference holder on the other hand. The common cooling rate 10  $^{0}$ C / min used, where the origin on the abscissa is the temperature. It is seen that the details of the hypereutectic AlSi alloys, the reaction discussed above are hardly distinguished on such integral curves. To get more insight into their observation, it would be of interest to use a more sophisticated model for DTA such as the one developed by Boettinger and Kattner [13] who took into account heat transfers between the sample & the cup and between the cup & the thermocouple. Thus; allowing an estimation of the temperature for true sample. Finally, in this research on the basis of the TGA and DTA traces during reactions for the hypereutectic AlSi following conclusions are made:

As per DTA results, the temperature  $680\,^{\circ}$ C is considered as the melting temperature of AlSi25 alloy. The melting value obtained for AlSi20 alloy, is higher than the previous (AlSi15) alloy, this is because of the higher contents of Si. This value is little lower than the actual value of the AlSi alloys and is because of selection of heating rate as  $10\,^{\circ}$ C / min. Any variation in the recorded value and actual value in the melting temperature is because of the heating rate [11].

For AlSi20 alloy, the endothermic reaction starts at 603 °C and ends at 661 °C, this cause the change in the crystalline phase to melting phase of the alloy and considered the melting temperature for the alloy as 661 °C.

As per TGA graph, the change in mass is 1.57 mg is recorded at the end of endothermic reaction. These both values are deviated from each other and are because of the different Si content. The value obtained through DTA traces for AlSi 20 is very near to the actual value of melting temperature of AlSi alloy (660 °C).

Whereas for  $\overline{AISi25}$  alloy; the endothermic reaction starts at 640 °C and ends at 680 °C and cause to change from crystalline phase to melting (liquid) phase of the alloy and considered the melting temperature for the alloy as 680 °C. As per TGA graph, the change in mass is 1.43 mg is recorded at the end of endothermic reaction. The observations were made to use very much related literature to furnish this research and on that basis, it can also be concluded that the heating rate for obtaining DTA and TGA is very important to find accurate values in the thermal analysis of the materials.

The authors wish to explore further this work to find the results related to determination of caloric values such as the heat of fusion or heat of crystallization and melting. This can be done with differential scanning calorimetry (DSC). DSC and DTA are both used in measuring a glass transition, phase changes, melting, purity crystallization, heat capacity, etc.

### Acknowledgement

This research work is sponsored by Research Management Center (RMC) of the Universiti Tun Hussein Onn Malaysia (UTHM) under Grant No. U872.

#### References

- [1] Lee JA. "Cast aluminum alloy for high temperature applications". NASM/Marshall Space Flight Center (MSFC) Materials. Mail Code ED33 Huntsville, AL, 358 12 USA; 2003. pp. 1–8.
- [2] Kasprzak, W., J. Sokolowski, H. Yamagata, M. Aniolek, and H. Kurita. "Energy Efficient Heat Treatment for Linerless Hypereutectic Al-Si Engine Blocks Made Using Vacuum HPDC Process". Journal of Materials Engineering and Performance. 2010. p. 1-13.
- [3] Paul Marchwica. "Microstructural and Thermal Analysis of AlSi and MgAl Alloys Subjected to High Cooling Rates". Master of Applied Science Thesis. University of Windsor, Ontario, Canada. 2012.
- [4] Hernandez FCR, Sokolowski JH. "Thermal analysis and microscopically characterization of Al–Si hypereutectic alloys". Journal of Alloys and Compounds. 419(1-2), 2006. pp. 180–190.
- [5] Aqeel Ahmed, M. S. Wahab, A. A. Raus, K. Kamarudin, Qadir Bakhsh and Danish Ali. "Mechanical Properties, Material and Design of the Automobile Piston: An Ample Review". Indian Journal of Science and Technology, Vol 9(36), 2016. DOI: 10.17485/ijst/2016/v9i36/102155.
- [6] Leonhard Heusler and Wolfgang Schneider. "Influence of alloying elements on the thermal analysis results of Al–Si cast alloys". Journal of Light Metals 2, 2002. pp. 17–26.
- [7] Ba"ckerud L, Chai G, Tamminen J. AFS/SKANALUMINIUM 71, 1990. pp. 229.
- [8] Samuel FH, Samuel AM, Doty HW. AFS Trans; 104, 1996. pp.893.
- [9] E.J. Martı'nez D, M.A. Cisneros G, S. Valtierra, J. Lacaze. "Effect of Strontium and Cooling Rate upon Eutectic Temperatures of A319 Aluminum Alloy". Scripta Materialia 52, 2005. pp. 439–443
- [10] Grega Klančnik1, Jožef Medved1, Primož Mrvar. "Differential thermal analysis (DTA) and Differential Scanning Calorimetry (DSC) as a Method of material Investigation". RMZ Materials and Geoenvironment, Vol. 57 (1), 2010. pp. 127–142.
- [11] A.M. Baghdasaryan, O.M. Niazyan, H.L. Khachatryan, S.L. Kharatyan. "DTA/TGA study of molybdenum oxide reduction by Mg/Zn & Mg/C combined reducers at non-isothermal conditions". Int. Journal of Refractory Metals and Hard Materials 51, 2015. pp. 315–323
- [12] Jacques Lacaze, Sonja Steinbach, Lorenz Ratke and Alexandre Freulon. "A DTA study of the solidification of Al-Si alloys". Proceedings of the 5th Decennial International Conference on Solidification Processing, Sheffield, July 2007.
- [13] W.J. Boettinger, U.R. Kattner, Metall. Mater. Trans. A, 33A, 2002. pp. 1779.
- [14] J. Lacaze, S. Ford, C. Wilson, E. Dubu, Scand. J. Metall., 22, 1993. pp. 300.