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Mechanical Property Comparison of Al11Si Wheels Grain Refined by Ti, Nb and MTS

 F. Aydogan ^a, K.C. Dizdar ^b , H. Sahin ^b , E. Mentese ^a, D. Dispinar ^{b,*} 
^a Doktas Wheels, Turkey^b Istanbul Technical University, Turkey

* Corresponding author. E-mail address: derya.dispinar@gmail.com

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Abstract

In this work, 25 wheels were cast with three different grain refiners: Al5Ti1B, Al3Nb1B and MTS 1582. Samples were machined from the wheels to check the mechanical properties. It was found that Nb grain refinement had the lowest grain size (260 mm) and highest tensile properties (yield strength of 119-124 MPa and ultimate tensile strength of 190-209 MPa). Al5Ti1B and MTS 1582 revealed quite similar results (110 MPa yield and 198 MPa ultimate tensile strength). The fading of the grain refining effect of Al5TiB1 master alloy was observed in both Nb and Ti added castings whereas during the investigated time interval, the fading was not observed when MTS 1582 was used.

Keywords: Al11Si, Grain refinement, Nb, MTS

Achievement of finer grains in casting of aluminum alloys known to have positive effects such as increased fluidity, increased feedability, decreased shrinkage, decreased porosity and increased mechanical properties. The process of grain refinement is the heterogeneous nucleation initiation by Al₃Ti intermetallic phases peritectically nucleates the Al-dendrites [1–4]. With regards to the Stoke's Law, the solid particles present in the liquid melt have the tendency to settle at the bottom of the crucible during casting operations known as the fading effect [5–8]. Limmaneevichitr [5] and Schaffer [7] showed that 20 minutes was enough to start the settlement process of TiB₂ and TiAl₃. Thus, the grain refinement efficiency decreases. In Al-Si alloys, there is also poisoning of Ti in the presence of Si which results in the formation of Si compounds on the surface of TiB₂ to inhibit the grain refinement [9–11]. Kori [12] and Ravi [13] on the other hand, proposed that higher B content overcomes the poisoning effect when Si content is higher than 7 wt%. Sigworth [8] suggested that high Ti content is needed in Al-Si alloys where minimum of 0.1 wt.% Ti is required. Timelli [14] looked into the grain refinement of recycled A356 alloy and concluded that addition of extra Ti to the recycled alloy had revealed no significant improvement in the grain size.

Several works have focused on alternative grain refiners, particularly rare-earth additions. Nowak [15–18] studied the use of Nb as grain refiner in aluminum alloys. It was reported that in the presence of B, Nb was an effective grain refiner in Al-Si alloys. Alternatively, Foseco developed a flux named Nucleant 1582 (MTS) that contains Ti- and B-bearing compounds (such as K₂TiF₆ and KBF₄) which leaves fresh nuclei of TiB₂ within the aluminium melt. These finely dispersed species are highly efficient nuclei that promote a fine equiaxed grain growth during solidification. Aydogan [19] had shown the statistical analysis of tensile property change of different grain refiners.

In this work, the effect of three different grain refiners on microstructure and mechanical properties of Al11Si alloy was investigated. Al3NbB and MTS 1582 additions were used as the grain refiner where the results were compared with the existing Al5Ti1B grain refined castings. All of the grain refiners were added before degassing. Degassing was carried out for 10 minutes with Foseco SMARTT system in 900 kg capacity transfer crucible. Nb content was targeted to achieve 0.1 wt.% in the alloy. For MTS 1582 trials, 450 g of the flux was added to the melt during degassing. The chemical composition of the starting melt is given



in Table 1. Total of 25 wheels were cast. Wheels were collected in the sequence of 5 parts. Baker test samples were collected for grain size measurements. Wheels were heat treated for the conditions of

540 °C solutionizing for 6 hours, quenched in water and aged at 145°C for 4 hours. Samples were collected from three regions: bore, spoke and rim; and subjected to tensile testing.

Table 1.

Chemical composition of the alloy used in the experiments (wt.%)

Si	Fe	Cu	Mn	Mg	Zn	Ni	Sr	Ti
10.794	0.128	0.0031	0.0039	0.140	0.010	0.004	0.037	0.090

In Figure 1, representative microstructure images of Al11Si alloy with different grain refiner additions is given. Barker test was applied to measure grain size of samples. Electrolytic etching with Barker reagent consisting of a 5% tetrafluoroboric acid in distilled water is used. The sample to be tested acts as an anode in a galvanic

cell, which removes material from the sample surface and an anodic layer can be formed. With the Barker method, under a polarized light, a colored representation of the grain structure of aluminum materials is achieved. ASTM E-112 method was used to measure the grain size.

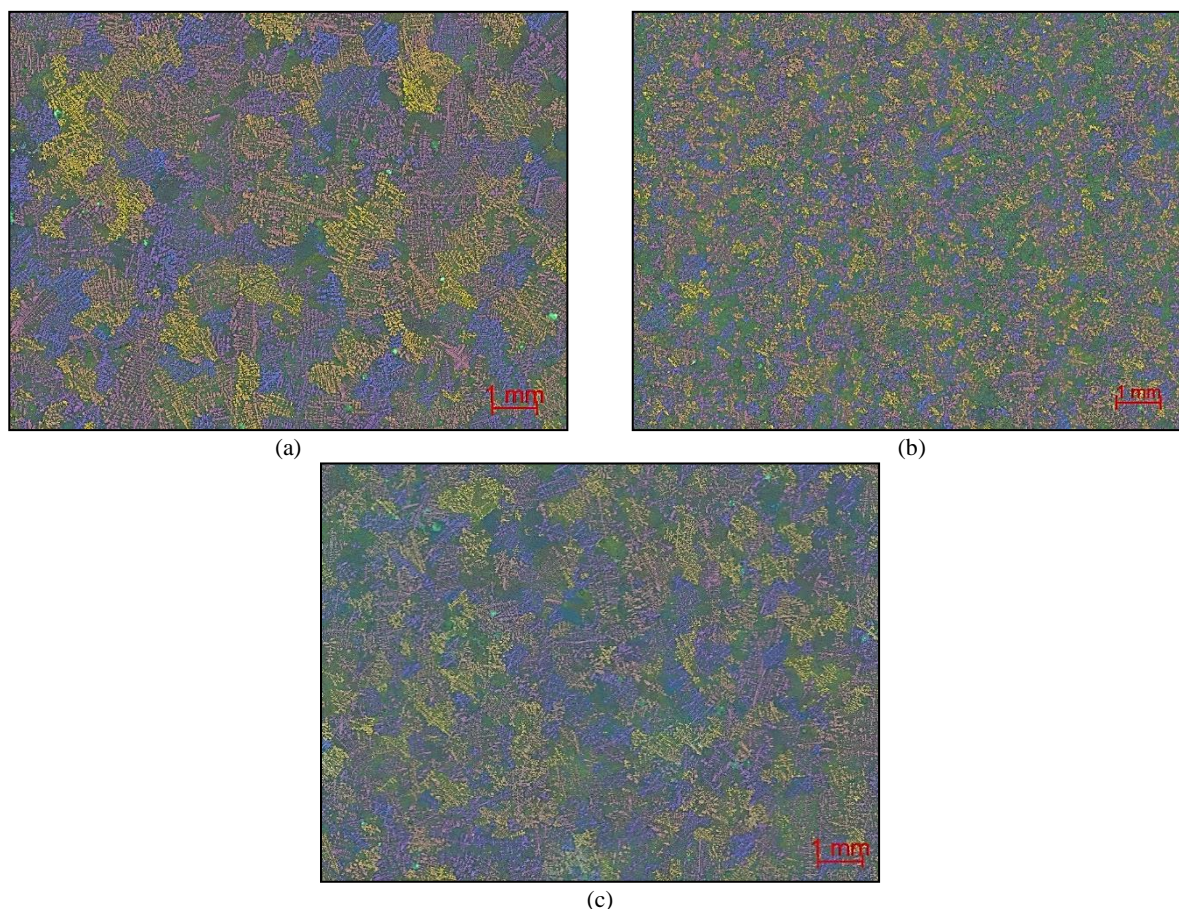


Fig. 1. Microstructural images of (a) Ti, (b) Nb, (c) MTS 1582 grain refined Al11Si alloy

As seen in Figure 2, Al₃NbB added castings had the lowest average grain size of 260 μm. This was followed by MTS 1582 where the average grain size was around 320 μm. On the other hand, Al₅TiB added castings revealed the highest grain size ranging between 350-550 μm with high scatter.

Nb master alloy contains 3 wt.% Nb and 1 wt.% B, therefore, during solidification NbB₂ and Al₃Nb is formed which are responsible for the heterogeneous nucleation of α-dendrites [20]. In Al₅Ti₁B master alloy, there are actually potentially three phases

that has the tendency to nucleate primary grains: Al₃Ti, TiB₂ and AlB₂. However, there is a long going debate in the literature as to which one has the active role in grain refinement. In both of these master alloys (Ti and Nb), these intermetallic phases exist already as a solid phase and the general approach is that they react peritectically to form the primary grain. On the other hand, MTS 1582 flux offers the formation of TiB₂ during degassing operation. Thus, the reaction of the flux with the molten aluminum results in the formation of TiB₂ which then nucleates the grain.

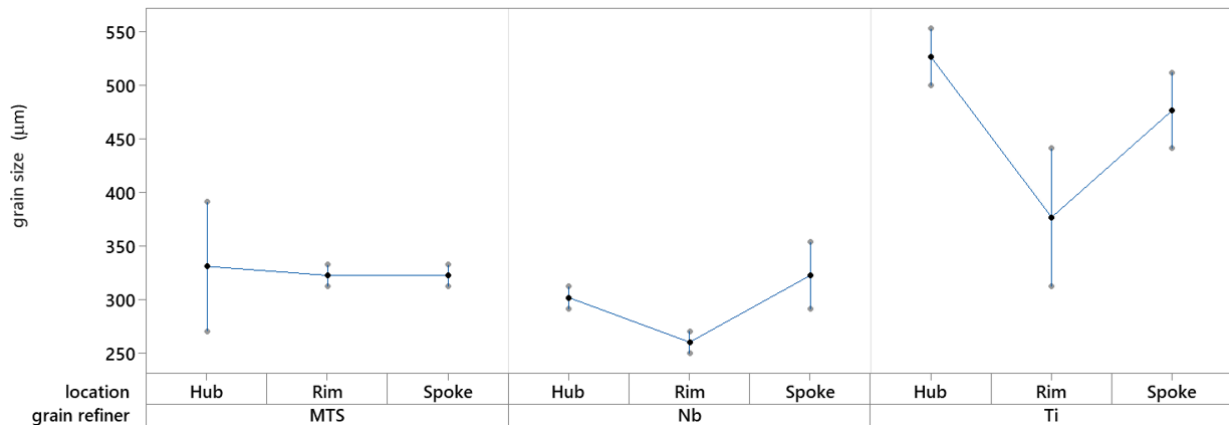


Fig. 2. Grain size change with different grain refiners

The yield strength of Al5Ti1B and MTS 1582 added melts revealed similar values as seen in Figure 3a which was around 110 MPa. Yield strength was not significantly changing by the location of the wheel. Al3NbB added wheels on the other hand, had the highest yield strength about 10-12 MPa higher than other additions. A similar scenario applies for the UTS values. As seen in Figure 3b, Al5Ti1B and MTS added melts had almost the same UTS value with Nb having the highest UTS for each section of the wheel. For

elongation at fracture values, Al3NbB reveals significantly higher values while, whereas, again, Al5Ti1B and MTS added melts had similar values (Fig 3c). The increase in mechanical properties is not just related with decreased size, but also the presence of dissolved and/or intermetallic phases in the matrix. In such cases, the resistance of dislocation movement appeared to be the highest in Al3NbB containing melt and thereby the tensile properties were higher for this alloy.

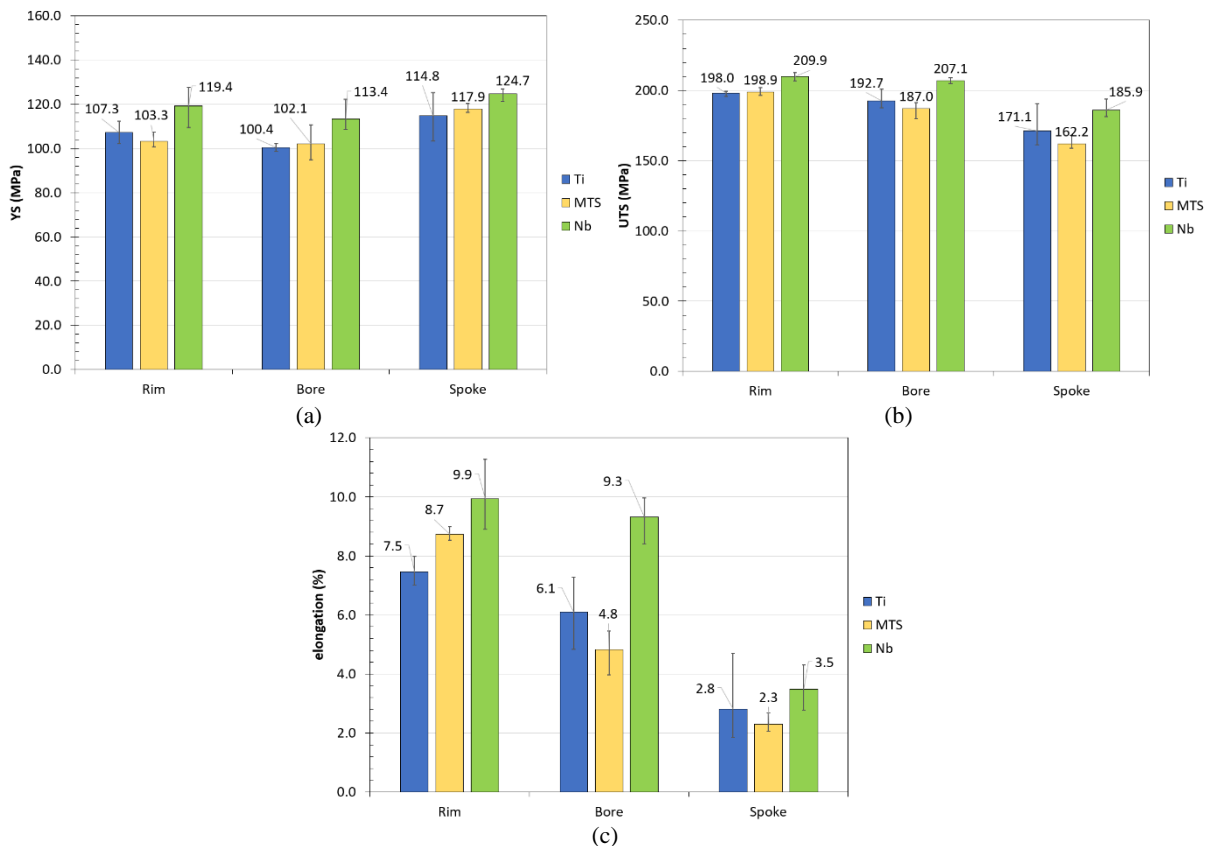


Fig. 3. Mechanical property change by section with different grain refiners: (a) yield strength, (b) ultimate tensile strength, (c) elongation at fracture

One of the interesting observations was the change in Ti content of the melt by the casting sequence. At every five wheels, an OES sample was collected from the furnace and the results are presented for all three grain refiners in Figure 4. It can be seen that Ti content was decreasing towards the end of the melt for Al5Ti1B and Al3Nb added melt. On the other hand, when MTS 1582 was

used, the Ti level was not changing and had remained constant through the casting process. This is mainly due to the fact that the working principle of MTS 1582 is based on in-situ nucleation of TiB₂ once the flux is reacted in the melt. On the other hand, TiB₂ has the tendency to sediment to the bottom of the furnace due to higher density [5-7].

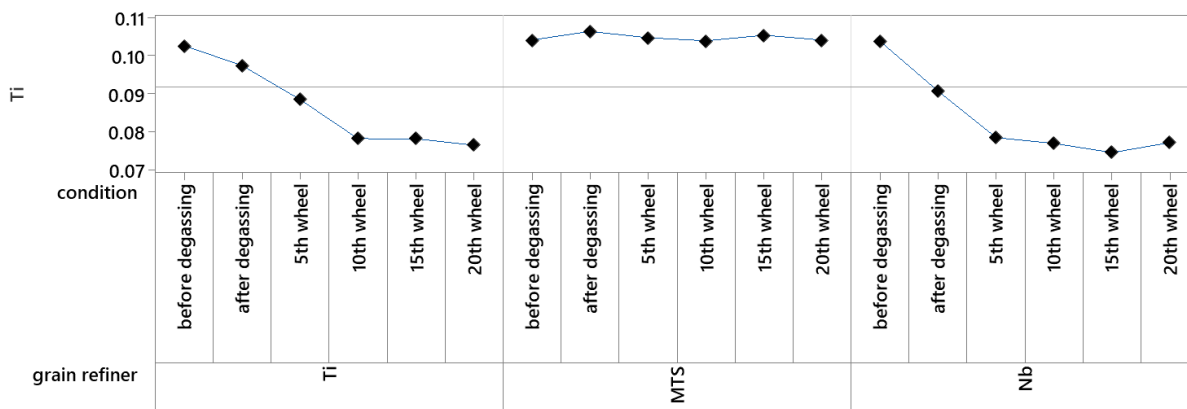


Fig. 4. Change in Ti content by casting sequence

As seen in Figure 5, during reaction of the MTS flux with the melt, much finer and homogeneously distributed grain refiners are introduced into melt (Fig 5, left), while coarser and agglomerated grain refiners start to sediment to the bottom of the crucible when Al5Ti1B is used (Fig , right).

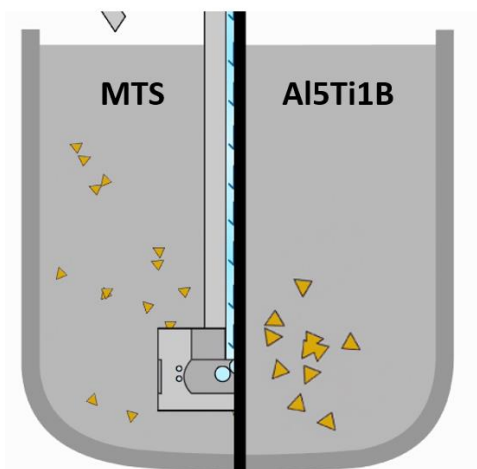


Fig. 5. Schematic representation of TiB₂ size and distribution in melt (Left: MTS, Right: Al5Ti1B; yellow triangles: TiB₂)

Nb grain refinement has significant effect on the microstructure with highest mechanical properties in Al11Si alloy. Al5Ti1B addition results in heterogeneous microstructure with decreasing grain refinement efficiency towards the end of the casting furnace. MTS 1582 exhibits lower grain size than Al5Ti1B addition. Fluxes are typically used in foundries for melt cleaning. MTS 1582 offers at the same time both cleaning of the melt and grain refinement of the microstructure and without any fading effect due to in-situ

nucleation of TiB₂. Additionally, MTS 1582 reveals similar tensile properties as Al5Ti1B addition.

Conflict of interest

On behalf of all authors, the corresponding author states that there is no conflict of interest.

References

- [1] Fan, Z., Wang, Y., Zhang, Y., Qin, T., Zhou, X., Thompson, G., Pennycook, T. & Hashimoto, T. (2015). Grain refining mechanism in the Al/Al-Ti-B system. *Acta Materialia*. 84, 292-304. <https://doi.org/10.1016/j.actamat.2014.10.055>
- [2] Schumacher, P., Greer, A., Worth, J., Evans, P., Kearns, M., Fisher, P. & Green, A. (1998). New studies of nucleation mechanisms in aluminium alloys: implications for grain refinement practice. *Materials Science and Technology*. 14, 394-404. <https://doi.org/10.1179/mst.1998.14.5.394>
- [3] Easton, M. & StJohn, D. (1999). Grain refinement of aluminum alloys: Part I. the nucleant and solute paradigms—a review of the literature. *Metallurgical and Materials Transactions*. A. 30, 1613-1623. <https://doi.org/10.1007/s11661-999-0098-5>
- [4] Easton, M. & StJohn, D. (1999). Grain refinement of aluminum alloys: Part II. Confirmation of, and a mechanism for, the solute paradigm. *Metallurgical and Materials Transactions*. A. 30, 1625-1633. <https://doi.org/10.1007/s11661-999-0099-4>
- [5] Limmaneevichitr, C. & Eidhed, W. (2003). Fading mechanism of grain refinement of aluminum-silicon alloy with Al-Ti-B grain refiners. *Materials Science and*

- Engineering: A.* 349(1-2), 197-206. [https://doi.org/10.1016/S0921-5093\(02\)00751-7](https://doi.org/10.1016/S0921-5093(02)00751-7)
- [6] Gürsoy, O., Erzi, E., & Dışpınar, D. (2019). Ti grain refinement myth and cleanliness of A356 melt. *Shape Casting*. Springer. 125- 130. https://doi.org/10.1007/978-3-030-06034-3_12
- [7] Schaffer, P.L. & Dahle, A.K. (2005). Settling behaviour of different grain refiners in aluminium. *Materials Science and Engineering: A.* 413, 373-378. <https://doi.org/10.1016/j.msea.2005.08.202>
- [8] Sigworth, G.K. & Kuhn, T.A. (2007). Grain refinement of aluminum casting alloys. *International Journal of Metalcasting. I.* 31-40. DOI:10.1007/BF03355416
- [9] Greer, A.L., Cooper, P.S., Meredith, M.W., Schneider, W., Schumacher, P., Spittle, J.A. & Tronche, A. (2003). Grain refinement of aluminium alloys by inoculation. *Advanced Engineering Materials.* 5, 81-91. <https://doi.org/10.1002/adem.200390013>
- [10] Li, Y., Hu, B., Liu, B., Nie, A., Gu, Q., Wang, J. & Li, Q. (2020). Insight into Si poisoning on grain refinement of Al-Si/Al-5Ti-B system. *Acta Materialia.* 187, 51-65. DOI: 10.1016/j.actamat.2020.01.039
- [11] Li, Y. Gu, Q.-F., Luo, Q., Pang, Y., Chen, S.-L., Chou, K.-C., Wang, X.-L. & Li, Q. (2016). Thermodynamic investigation on phase formation in the Al-Si rich region of Al-Si-Ti system. *Materials and Design.* 102, 78-90. <https://doi.org/10.1016/j.matdes.2016.03.144>
- [12] Kori, S., Murty, B. & Chakraborty, M. (1999). Influence of silicon and magnesium on grain refinement in aluminium alloys. *Materials Science and Technology.* 15, 986-992. <https://doi.org/10.1179/026708399101506823>
- [13] Ravi, K., Manivannan, S., Phanikumar, G., Murty, B. & Sundarraj, S. (2011). Influence of Mg on grain refinement of near eutectic Al-Si alloys. *Metallurgical and Materials Transactions A.* 42, 2028-2039. DOI: 10.1007/s11661-010-0600-0
- [14] Timelli, G., Camicia, G. & Ferraro, S. (2014). Effect of grain refinement and cooling rate on the microstructure and mechanical properties of secondary Al-Si-Cu alloys. *Journal of Materials Engineering and Performance.* 23(2), 611-621. DOI:10.1007/s11665-013-0757-y
- [15] Nowak, M., Bolzoni, L. & Babu, N.H. (2015). Grain refinement of Al-Si alloys by Nb-B inoculation. Part I: Concept development and effect on binary alloys. *Materials & Design.* 1980-2015. 66, 366-375. <https://doi.org/10.1016/j.matdes.2014.08.066>
- [16] Nowak, M., Yeoh, W., Bolzoni, L. & Babu, N.H. (2015). Development of Al-Nb-B master alloys using Nb and KBF4 powders. *Materials & Design.* 75, 40-46. <https://doi.org/10.1016/j.matdes.2015.03.010>
- [17] Bolzoni, L., Nowak, M. & Babu, N.H. (2015). Grain refinement of Al-Si alloys by Nb-B inoculation. Part II: application to commercial alloys. *Materials & Design.* 1980-2015. 66, 376-383. <https://doi.org/10.1016/j.matdes.2014.08.067>
- [18] Bolzoni, L., Nowak, M. & Babu, N.H. (2015). On the effect of Nb-based compounds on the microstructure of Al-12Si alloy. *Materials Chemistry and Physics.* 162, 340-345. <https://doi.org/10.1016/j.matchemphys.2015.05.076>
- [19] Aydogan, F., Dizdar, K.C., Sahin, H., Mentese, E. & Dispinar, D. (2022). Weibull analysis evaluation of Ti, B, Nb and MTS grain refined Al11Si alloy. *Materials Chemistry and Physics.* 126264. <https://doi.org/10.1016/j.matchemphys.2022.126264>
- [20] Xu, J., Li, Y., Hu, B., Jiang, Y. & Li, Q. (2019). Development of Al-Nb-B master alloy with high Nb/B ratio for grain refinement of hypoeutectic Al-Si cast alloys. *Journal of Materials Science.* 54(23), 14561-14576. DOI:10.1007/s10853-019-03915-9