Official URL: https://doi.org/10.1007/s12666-018-1426-5

To cite this version:

Ferdian, Deni and Irawan, K. Saputra and Lacaze, Jacques *Effect of Cooling Rate and Neodymium Addition on Beta Intermetallic Phase of Al–Fe–Si Ternary System.* (2018) Transactions of the Indian Institute of Metals, 71 (11). 2671-2676. ISSN 0972-2815

Effect of Cooling Rate and Neodymium Addition on Beta Intermetallic Phase of Al–Fe–Si Ternary System

D. Ferdian[1] · K. Saputra Irawan[1] · J. Lacaze[2]

Abstract Aluminium silicon alloys are widely used in automotive industry and other structural application. However, the presence of high content of iron element in Al–Si alloys lead to precipitation of beta intermetallic phase that has a detrimental effect on mechanical properties. Reducing the adverse effects of β-$Al_9Fe_2Si_2$ precipitates can be achieved by altering their morphology by adding element modifier and increasing solidification cooling rate. In this present work, simultaneous thermal analysis was used to study the effect of cooling rate (5, 10 and 30 °C min^{-1}) on beta phase formation in Al–7Si–1Fe alloy added with neodymium at 0.3, 0.6 and 1 wt%. The beta phase precipitates were then characterized using optical microscopy and scanning electron microscopy equipped with EDS. Image analysis results showed the reduction in size of beta intermetallic phase as a result of the rare earth addition. Further analysis also showed the refinement of eutectic silicon.

Keywords Neodymium · Cooling rate · Beta intermetallic phase · Al–Si alloys

✉ D. Ferdian
deni@metal.ui.ac.id

1 Department of Metallurgy and Materials Engineering, Universitas Indonesia, Kampus Baru UI, Depok 16424, Indonesia

2 CIRIMAT, Université de Toulouse, ENSIACET, BP 44362, 31030 Toulouse Cedex 4, France

1 Introduction

Aluminium silicon alloys are mainly used due to their superiority in manufacturing process especially lightweight characteristic, casting fluidity and heat treatability with magnesium or copper addition. Nevertheless, in the presence of impurities such as iron element, it will form iron-rich intermetallic phase such as beta phase (β-$Al_9Fe_2Si_2$) which is detrimental to the mechanical properties. The formation of beta phase is highly influenced by the cooling process of solidification [1–4], and some elements such as manganese favour the formation of alpha cubic α-$Al_{15}(Fe,Mn)_3Si_2$ phase instead of beta phase [5]. Also, rare earth elements are known to modify the eutectic silicon [6–8]. Furthermore, other studies show their influence on the morphology of beta phase as seen in the work by Li et al. [6] who used La and Y and Rao et al. [8] with samarium. Nevertheless, the use of neodymium as beta phase modifier has not been reported. Therefore, the objective of this present work is to characterize the effect of neodymium (Nd) on the beta phase morphology in high purity Al–Fe–Si ternary system.

2 Experimental Procedures

High purity (99.5%) Al ingot, Al–Si alloy (made from Al ingot and 443 Si grade) and Al–Fe alloy (made from Al ingot and 80 Fe—Hoesch alloying tablet) were used to synthetize an Al–7Si–1Fe alloy by melting in a graphite crucible under ambient air. The chemical composition of the master alloy is listed in Table 1. Rare earth element was added using a high purity binary Al–Nd alloy to achieve the desired content of 0.3, 0.6 and 1 wt% Nd. The melting furnace being set at 720 °C, the molten metal was

Table 1 Chemical composition of Al–7Si–1Fe alloys (wt%)

Si	Fe	Cu	Mn	Mg	Ti	Sr	Ni	Al
7.20	1.19	0.0014	< 0.001	0.0024	0.0105	< 0.0001	0.0005	Bal.

then stirred using a graphite bar and held for 30 min to ensure homogenization. Finally, the slag was removed prior pouring into the mould. Unfortunately, Nd addition could not be measured due to limitation with the optical emission spectrometer apparatus that was used.

The alloy was then subjected to simultaneous thermal analysis with a PerkinElmer STA6000 apparatus for differential scanning calorimetry (DSC) analysis. The runs were performed with cooling rate of 5, 10 and 30 °C min^{-1} under nitrogen. The DSC samples were then prepared for metallographic observation by grinding with abrasive papers of decreasing size and finally polishing with 0.05-μm alumina suspension in alcohol. Metallographic analysis was carried out using optical microscopy (Olympus BX 51) and scanning electron microscopy (FEI Inspect F50) equipped with energy-dispersive spectrometry (EDAX). Moreover, 2D image analysis of backscattered electrons (BSE) was carried out using imageJ software [9] for beta phase length analysis.

3 Result and Discussion

Figure 1 illustrates the DSC thermograms for the three selected cooling rates in the case of the alloy without Nd addition. DSC thermograms show a three-step solidification reaction consisting of nucleation and growth of primary (Al) dendrites (a), then precipitation of the beta phase (b) and finally of (Al)-silicon eutectic (c). A shifting of the thermal arrests to lower the temperature is observed as the scanning rate increases.

Figure 2 compares the DSC thermograms recorded at 30 °C min^{-1} for the alloys without and with Nd additions. Addition of Nd slightly shifts the temperature for beta phase formation from 566 to 565 °C. A shift is also noticed for the (Al)–Si eutectic from 552 °C without Nd to 548 °C with 1 wt% Nd.

Microstructure of the samples observed by optical microscopy shows the presence of the (Al) matrix, beta phase precipitates and eutectic silicon. The influence of neodymium addition for a scanning rate of 30 °C min^{-1} can be seen in Fig. 3. The beta phase in white contrast appears shorter on average as the neodymium content increases. EDS mapping and micro-chemical analysis as seen in Figs. 4 and 5 indicate the formation of an intermetallic compound containing neodymium. The composition of this intermetallic compound (point 6 in Fig. 5) can correspond to the formula $AlNd_2Si_2$, in agreement with Al–Nd–Si phase diagram information [10].

Measurements of beta phase length—both the average length and the mean of the five longest particles—are

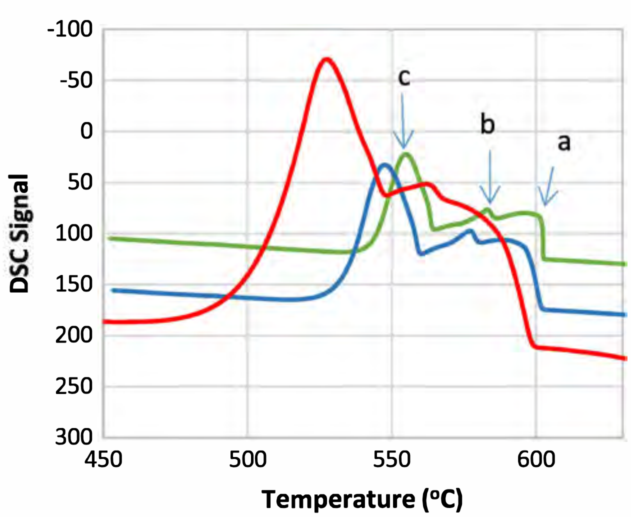


Fig. 1 DSC thermograms upon cooling at various scanning rates (green line: 5 °C min^{-1}; blue line: 10 °C min^{-1}; red line: 30 °C min^{-1})

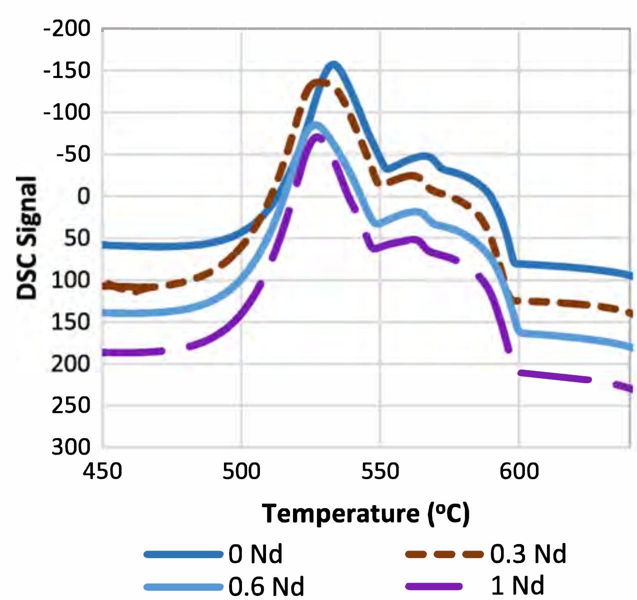


Fig. 2 DSC thermograms upon cooling at 30 °C min^{-1} for various neodymium additions

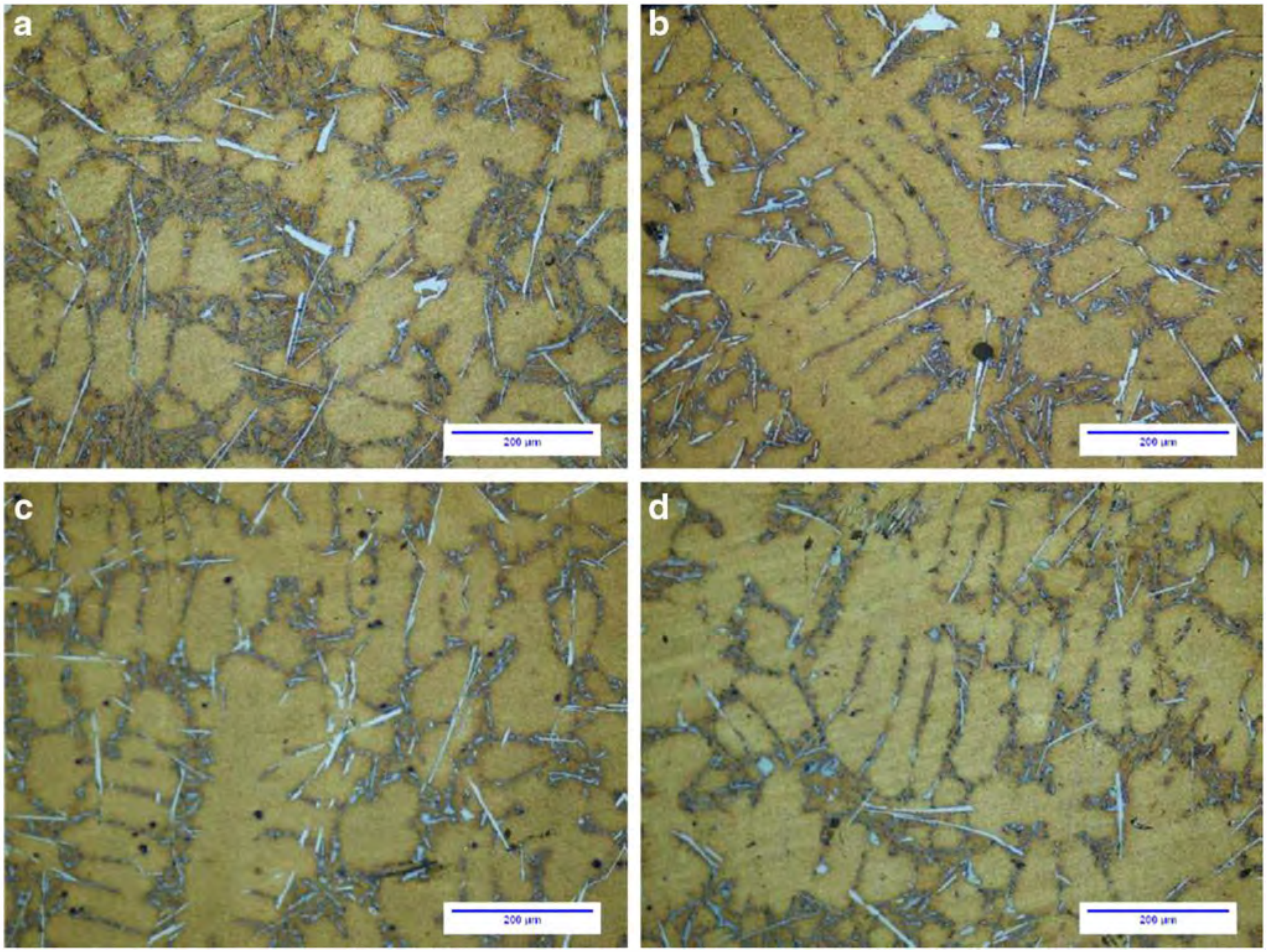


Fig. 3 Optical micrograph of DSC samples with various neodymium additions: **a** 0%, **b** 0.3%, **c** 0.6%, and **d** 1%

shown in Fig. 6. Increasing the neodymium content reduces the maximum length of the five largest particles from 290 µm without Nd addition to 206 µm with 1%wt Nd, i.e. approximately 30% reduction. At the same time, the average beta phase length decreases from 202 to 145 µm. Figure 6 also shows the length distribution of the beta phase, where the addition of Nd shifts the beta phase length distribution to a lower range. Without any addition of Nd, the minimum length of beta particles is over 150 µm and 20% of the beta precipitates are over 250 µm. By adding 1% Nd, very few beta particles are larger than 200 µm and 13% have a size below 100 µm. The size reduction mechanism by Nd addition can be promoted by the blocking the Si and Fe atoms during the beta phase growth.

Furthermore, the addition of Nd reduces the length of eutectic silicon. Increasing the neodymium reduces the average length of eutectic silicon from 35 µm without any addition to 14 µm with 1% Nd addition as illustrated in Fig. 7. This reduction can be explained by impurity-induced twinning mechanism as originally suggested by Lu

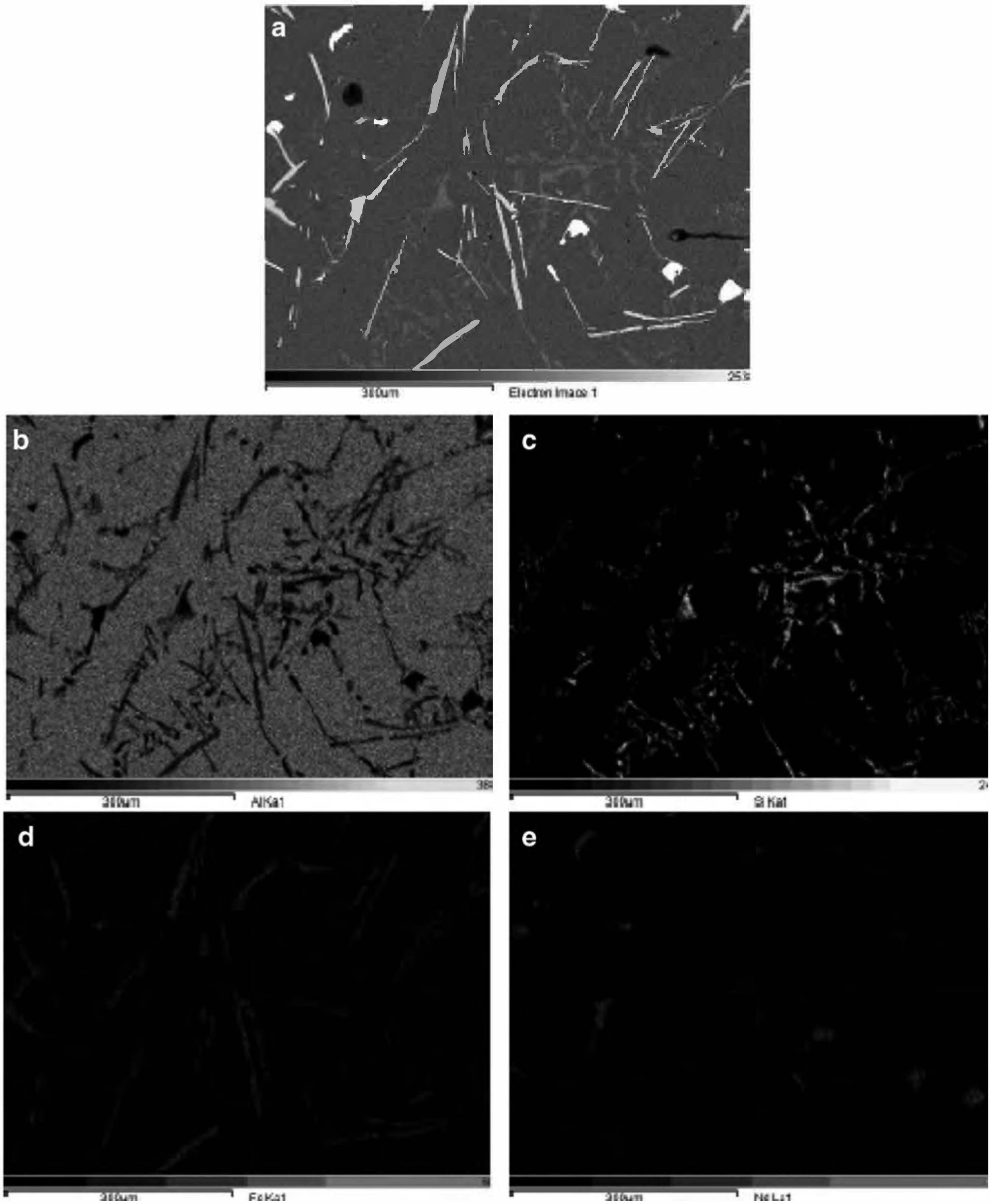
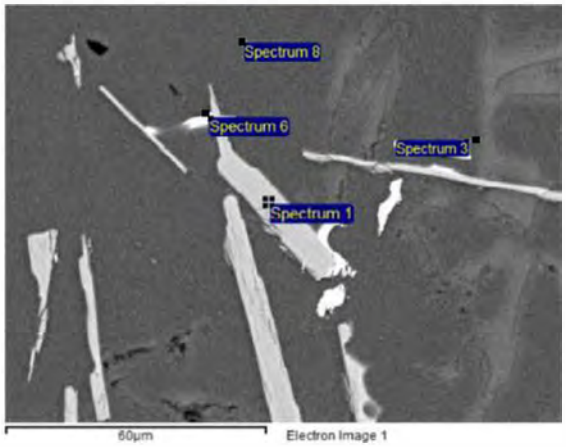


Fig. 4 Backscattered electrons SEM micrograph of Al–7Si–1Fe–1Nd alloy (**a**) and related EDS mappings of aluminium (**b**), silicon (**c**), iron (**d**) and neodymium (**e**)



No	(atomic %)				Phase identification
	Al	Si	Fe	Nd	
Point 1	66.3	19.1	14.6	-	$Al_9Fe_2Si_2$
Point 3	10.0	90.0	-	-	Si
Point 6	98.5	1.2	0.04	0.04	AlSiNd
Point 8	98.5	1.5	-	-	(α)-Al

Fig. 5 EDS micro-analysis of the phases in Al–7Si–1Fe–1Nd alloy taken from BSE image

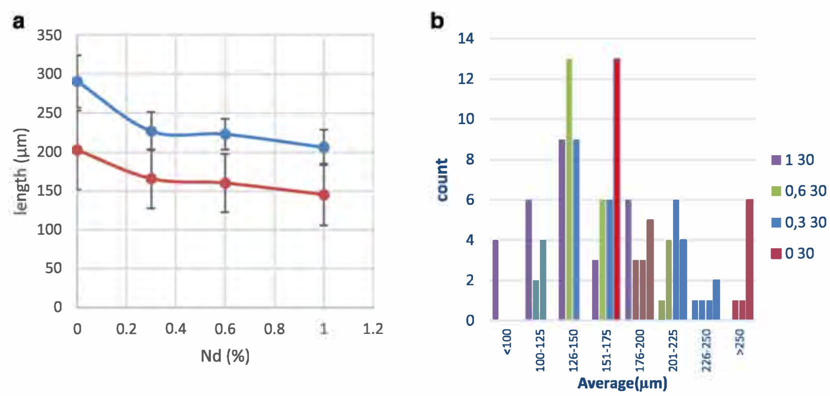


Fig. 6 Evolution with the Nd content of beta phase length in DSC samples cooled at 30 °C min^{-1}: **a** beta phase maximum and average length (blue line: maximum, red line: average). **b** Distribution of beta phase length

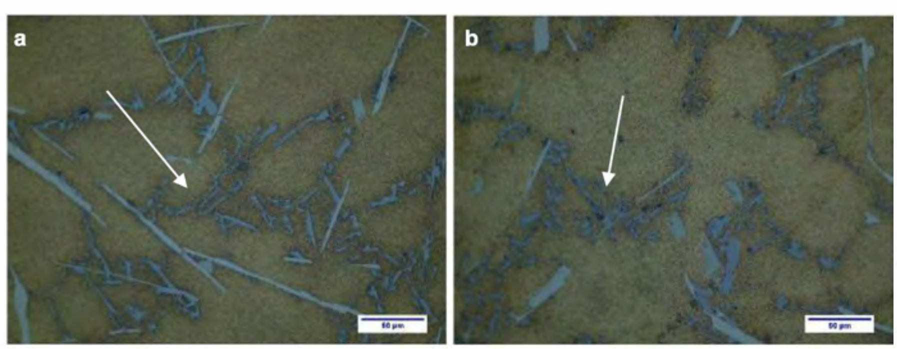


Fig. 7 Optical micrograph of DSC samples cooled at 30 °C min^{-1} showing the reduction in size of eutectic silicon (white arrow) with neodymium additions: **a** 0% and **b** 1%

and Hellawell [11] and confirmed more recently by Timpel et al. [12] and Barrirero et al. [13]. According to these authors, the twins grow when the atomic size ratio of the modifier element with silicon is high enough, which is the case with Nd element for which the atomic size ratio is 1.55.

4 Conclusion

Rare earth addition to an Al–Fe–Si alloy reduces the beta phase average length. Reduction achieved amounts to 20% with 1% Nd addition (from 202 µm to 145 µm). The results also show the refinement of eutectic silicon with Nd addition.

References

1. Samuel A M, Samuel F H, and Doty H W, *J Mater Sci* **31** (1996) 5529.
2. Khalifa W, Samuel F H, and Gruzleski J E, *Metall Mater Trans. A* **34** (2003) 807.
3. Ferdian D., Suharno B., Duployer B, Duployer B, Tenailleau C, Salvo L, and Lacaze J, *Trans Indian Inst Met* **65** (2012) 821.
4. Gorny. A, Manickaraj J, Cai Z, and Shankar B, *J Alloys Compd*, **577** (2013) 103.
5. Munson D, *J Inst Met*, **95** (1967) 217.
6. Li Z, and Yan H, *J Rare Earths* **33** (2015) 995.
7. Ghoncheh MH, Shabestari SG, and Abbasi MH, *Therm Anal Calorim* **117** (2014) 1253.
8. Rao Y, Yan H, and Hu Z, *J Rare Earths* **33** (2013) 916.
9. Schneider C A; Rasband W S, and Eliceiri K W, *Nature methods* **9** (2012) 671.
10. Raghavan V, *JPEDAV* **33**, 920120, 66.
11. Lu S Z, and Hellawell A. *Metall Trans A* **18** (1987) 1721.
12. Timpel M, Wanderka N, Schlesiger N, Yamamoto T, Lazarev N, Isheim D, Schmitz G, Matsumura S, and Banhart J, *Acta Mater* **60** (2012) 3920.
13. Barrirero J, Engstler M, Ghafoor N, de Jonge N, Odén M, and Mücklich F, *J Alloys Compd* **611** (2014) 410.