THERMAL EFFECT OF QUENCH RATE ON MECHANICAL PROPERTIES OF SIMA 7075-T6

by

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In this study, thermal effects of quench rate on mechanical properties of 7075-T6 alloy fabricated by strain induced melt activation (SIMA) process were investigated. The T6 heat treatments were applied in various parameters after SIMA: solutionizing at 490 °C for 15-30-45-60-120 minutes, quenching at 60-100 °C, and aging at 120 °C for 12-24-48 hours. Optical and SEM analyses were performed for characterization, hardness, and tensile tests for mechanical evaluation. Analysis of variance tests was carried out to indicate the validity of the interactions quenching temperatures and tensile properties. Secondary phases were dissolved homogeneously after solutionizing but at higher quenching temperatures and re-precipitated, particularly at grain boundaries. Strength values decreased with increasing quenching temperature. Analysis of variance tests suggest that cryogenic quenching can further increase the tensile properties. Oxide bifilms, porosities, low surface quality, and continuous brittle secondary phases surrounding spheroidal particles reduce the mechanical properties.

Key words: *aging, aluminum alloys, electron microscopy, analysis of variance, semisolid*

Introduction

Since the beginning of the 1970's, several semisolid forming processes such as thixocasting, thixoforging, thixomolding, rheocasting, and rheomolding have been developed [1, 2]. Today, semisolid forming has become a commonly used manufacturing technology for the production of aluminum and magnesium alloy parts that combines the advantages of conventional manufacturing methods such as forging in solid state and casting in liquid state [3-5]. The SIMA process is among the most established methods to achieve a homogeneous spheroidal grain structure required for semisolid forming [6-8]. The process is based on obtaining spheroidal grain structure by applying hot and cold deformation after casting then reheating to the semisolid temperature.

The 7075 alloy is used in areas requiring mechanical strength, lightweight and corrosion resistance in the aviation and automotive industries [9, 10]. Its wide solidification range of about 160 °C makes this alloy a good candidate for semisolid forming processes [5].

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The 7075 alloy must undergo age hardening treatment to achieve maximum strength after semisolid forming processes. However, different manufacturing techniques result in specific microstructures requiring different heat treatment parameters. Likewise, in SIMA process, large compositional differences occur between the spheroidal grains and grain boundaries and the resulting microstructure is very different from the as-extruded or as-cast microstructures. The effect of this unique microstructure on the selection of the precipitation hardening parameters for semisolid formed aluminum alloys remains unclear. To the knowledge of the authors, there is only few studies reported in the literature about T6 treatment of aluminum alloys manufactured by semisolid forming processes. In the study of Tan *et al.* [11] T6 heat treatment of SIMA 7075 alloy was carried out by solutionizing at 480 °C for 30 minutes, quenching in water of 20 °C and 80 °C and aging at 120 °C for 7 hours. Better mechanical properties (448.4 MPa tensile strength and 12.7% elongation) were reported when quenching was done at 80 °C. In the study of Mahathaninwong *et al.* [12] solutionizing at 450 °C and 480 °C for 1 to 12 hours and quenching in water at 25 °C and finally aging at various temperatures between 120 °C and 185 °C were applied to rheocast 7075 alloy. Optimum T6 treatment parameters for the rheocast 7075 alloy were reported as solutionizing at 450 °C for 4 hours and aging at 120 °C for 72 hours resulting 486 MPa tensile strength and 2% elongation.

The optimum SIMA process parameters to achieve the near spherical grain structure required for semisolid forming was determined in the previous study by the authors [13]. The aim of this study is to evaluate the effects of quench rate on mechanical and microstructure properties of 7075-T6 alloy fabricated by SIMA process.

Material and method

In this paper, 7075 aluminum alloy fabricated by extrusion followed by cold deformation, with the chemical composition in tab. 1 was used.

Table 1. Chemical analysis of the 7075 alloy [%]

Al	Zn	Mg	Cu	Fe	Si	Cr	Mn	Ni	Zr	Pb	Ti	Sn
90.3	5.08	1.97	1.21	0.5	0.26	0.23	0.21	0.04	0.04	0.02	0.02	0.018

The samples were heated up to the semisolid temperature (isothermal holding at 630 °C for 25 minutes) and water quenched at 20 °C. After SIMA process, samples were solutionized at 490 °C for 15-30-45-60-120 minutes and quenched in water at 60-100 °C. After determination of optimum solutionizing parameters, aging heat treatments at 120 °C for 12-24-48 hours were carried out.

Specimens were prepared by sectioning in appropriate size and grinding with 400, 1000, and 2400 grit SiC sandpapers and polishing with a 3 μm sized diamond paste in accordance with ASTM E3-11 (2017). Keller's solution (5 mL HNO_3, 3 mL HCl, 2 mL HF, and 100 mL H_2O) was used to investigate the microstructural evolution of the alloy.

Cylindrical tensile test samples were prepared from aged specimens into according to ASTM E8/E8M-16a by machining. The 15 tensile tests were carried out for each quench temperature. The SEM analyses were carried out on the fractured surface of the tensile test samples. Brinell hardness tests were carried out in accordance with ASTM E10-17 after each heat treatment and five measurements were taken for each sample to obtain an average hardness.

The ANOVA analysis was carried out to evaluate the relationship between quenching temperature and tensile properties.

Results and discussion

Homogeneous spheroidal grain structure suitable for semisolid forming consisting of solid α-Al grains and liquid phase around them is obtained by isothermal heat treatment at 630 °C for 25 minutes. Atkinson *et al.* [4] had shown that below 560 °C, the microstructure remains predominantly unrecrystallised due to grain boundary pinning and it takes longer times for partial remelting. Above this temperature, within 10 minutes, the aging precipitates starts to melt thereby unpinning the boundaries which results in equiaxed recrystallized (spherical) microstructures.

Large compositional differences can be seen between the spheroidal grains and grain boundaries containing higher amounts of alloying elements. Binesh and Aghaie-Khafri [7] reported that with 55% deformed samples, 15-25 minutes at 610 °C was good enough to achieve spherical grains. A detailed investigation of microstructural evolution of 7075 alloy during SIMA process can be found in the previous study by the authors [13]. After SIMA process, five samples were solutionized at 490 °C for 15-30-45-60-120 minutes and quenched in water at 20 °C. From the micrographs of the samples, it can be seen that the secondary phases existing in the grain boundaries dissolve after 45 minutes at 490 °C, fig. 1.

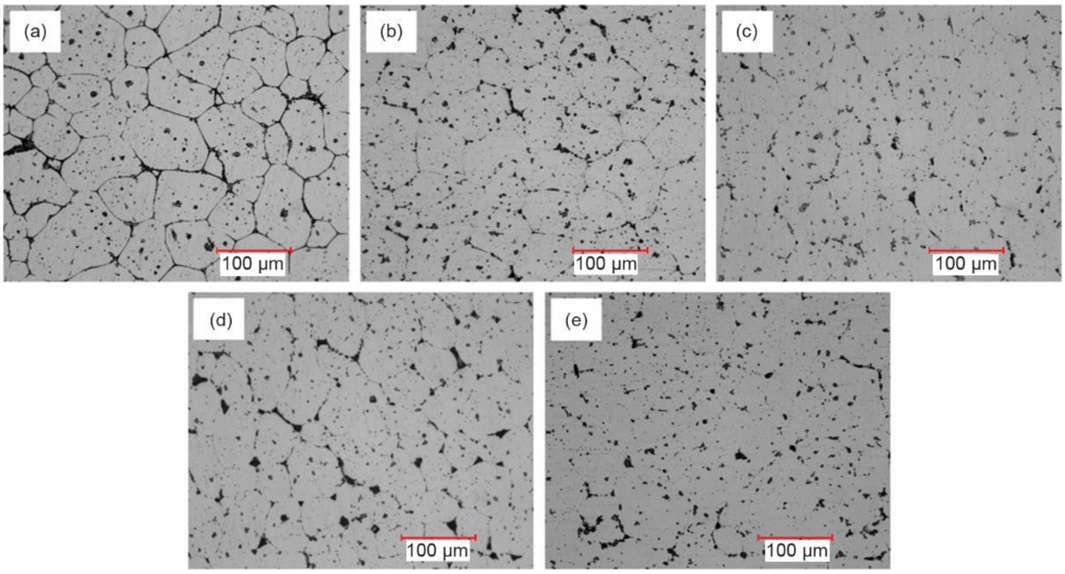

Figure 1. Micrographs of the samples solutionized at 490 °C for: (a) 15 minutes, (b) 30 minutes, (c) 45 minutes, (d) 60 minutes, and (e) 120 minutes

After determination of the optimum solutionizing duration, aging at 120 °C for 12-24-48 hours was carried out with nine samples, tab. 2. At first, spheroidizing by isothermal holding at 630 °C for 25 minutes at was applied to the samples. Then samples were separated into three groups and the solutionizing heat treatments were carried out with optimum parameters (490 °C, 45 minutes). The first group was quenched in water at 60 °C and the second group at 100 °C without allowing the samples to cool. Three samples from each group was aged at 120 °C for 12 hours, 24 hours, and 48 hours, respectively.

The evolution of hardness of the samples quenched at different temperatures and aged at 120 °C are shown in fig. 2. Since the maximum hardness values were obtained with all three quenching temperatures at 48 hours, it was chosen as the optimum aging time at 120 °C. It is important to note that as the soaking time was increased, coarsening could occur. Jiang *et al.* [6] reported that RAP microstructure gives higher coarsening than SIMA.

Table 2. Heat treatments applied to aging samples

Spheroidizing	Solutionizing [minutes]	Quenching in water [°C]	Aging at 120 °C [hours]
630 °C, 25 minutes	490 °C, 45 minutes	60	12
			24
			48
		100	12
			24
			48

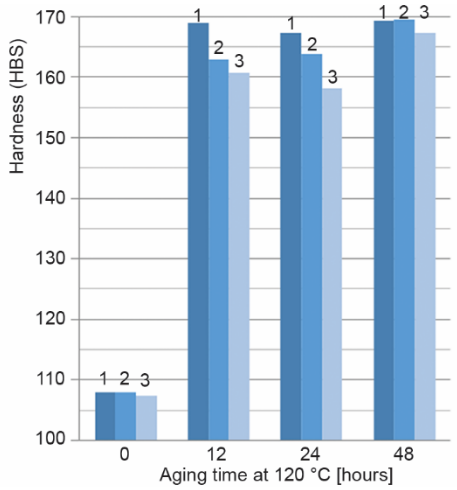

Figure 2. Hardness measurements after quenching and aging [13]; *1 – quenching at 20 °C, 2 – quenching at 60 °C, and 3 – quenching at 100 °C*

Mahathaninwong *et al.* [12] found that optimum solution treatment for semisolid 7075 alloy was 450 °C for four hours in which the eutectic phase was completely dissolved and no overheated Mg_2Si particles were formed. The highest hardness was achieved 120 °C for 72 hours. However, in this work, it was found that the heat treatment cycle required much lower durations. After determination of optimum aging time at 120 °C, tensile test specimens were subjected to spheroidizing (630 °C, 25 minutes), solutionizing (490 °C, 45 minutes), water quenching at 60 and 100 °C and precipitation (120 °C, 48 hours), tab. 3.

Table 3. Heat treatments applied to aging samples

Spheroidizing	Solutionizing	Quenching in water [°C]	Aging
630 °C, 25 minutes	490 °C, 45 minutes	60	120 °C, 48 hours

The main purpose of the quenching experiments performed at different temperatures was to examine the effect of the internal stresses generated after quenching on the mechanical properties of the material. It was observed that the quenching temperature after the solutionizing stage of the T6 heat treatment significantly affected the mechanical properties. This effect was observed mainly in yield strength and elongation after fracture values while there was no significant difference in hardness values. As a result of the tensile tests, the yield strength values obtained at different quenching temperatures are given in fig. 3.

It was observed that the yield strengths of the samples were increased from 225 MPa to an average of 533 MPa after heat treatment. The difference more than two folds is remarkable. In the previous study of the authors [13], while the maximum yield strength was reached at 20 °C quenching temperature with 546 MPa, in this study, maximum yield strength was obtained at 60 °C quenching temperature. Yield strength changes to 576 and 461 MPa with increasing quenching temperature to 60 and 100 °C, respectively. Mahathaninwong *et al.* [12] had reached 371 MPa with solutionizing at 450 °C for 4 hours, but the samples were quenched in room temperature.

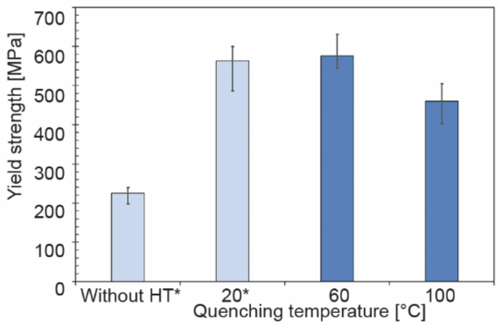

Figure 3. Yield strength values obtained at different quenching temperatures [13]

Based on the data obtained from tensile tests, ANOVA analysis, tab. 4, was carried out and the relationship between quenching temperature and yield strength was obtained:

$$y = -1.3408x + 614.23R^2 = 1 \quad (1)$$

where y is the yield strength and x – the quenching temperature. It can be understood from eq. (1) that as the quenching temperature is increased, yield strength will decrease.

This relationship, which is seen in the yield strength, is similar in the ultimate tensile strength. The average tensile strength of the samples increased from 381 MPa to 616 MPa after T6 treatment with 20 °C quenching temperatures [13], 627 and 545 MPa with 60 and 100 °C, respectively, fig. 4.

Table 4. The ANOVA test results comparing the relationship between yield strength and quenching temperature

	df	SS	MS	F	Significance F			
Regression	1.00	77033.24	77033.24	48.40	0.00			
Residual	39.00	62077.91	1591.74					
Total	40.00	139111.15						
	Coefficients	Standard error	t-Stat	P-value	Lower 95%	Upper 95%	Lower 95.0%	Upper 95.0%
Intercept	614.23	13.64	45.05	0.00	586.65	641.81	586.65	641.81
20.00	–1.34	0.19	–6.96	0.00	–1.73	–0.95	–1.73	–0.95

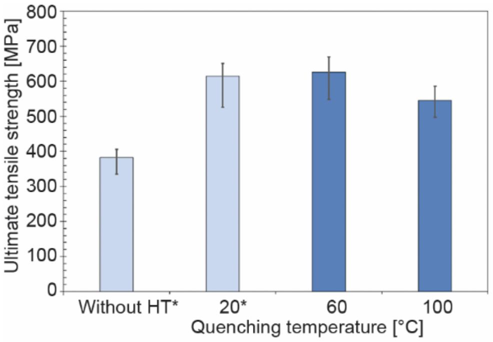

Figure 4. Ultimate tensile strength values obtained at different quenching temperatures [13]

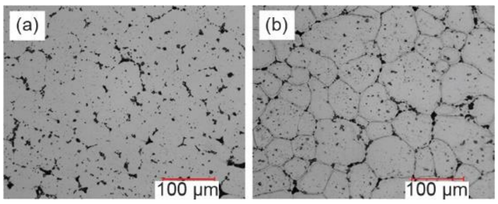

Figure 5. Micrographs of the samples quenched at: (a) 60 °C and (b) 100 °C

The reason for the decrease in strength is thought to be the decrease in the cooling rate due to the increased quenching temperature, providing enough time for the secondary phases that are homogeneously dissolved in the material while solutionizing, to precipitate into coarse particles. These coarse precipitates can be seen especially at the grain boundaries of the samples that are quenched at 100 °C, fig. 5.

This is mainly due to formation of less vacancy by increased quenching temperature. Although residual stress decreases in this case, it appears that the size and distribution of secondary phase plays more significant effect on mechanical properties. The ANOVA test results comparing the relationship between ultimate tensile strength and quenching temperature were given in tab. 5. according to:

$$y = -0.9057x + 650.48R^2 = 1 \quad (2)$$

where y is the ultimate tensile strength and x – the quenching temperature. Similar to findings of yield strength, as the quenching temperature was increased from 60 °C to 100 °C, the tensile strength decreases. However, it is interesting to note that the potential maximum ultimate tensile strength value for the alloy appears to be around 650 MPa. This also suggests that cryogenic quenching can further increase the tensile properties [14].

Table 5. The ANOVA test results comparing the relationship between ultimate tensile strength and quenching temperature

	df	SS	MS	F	Significance F			
Regression	1.00	35150.83	35150.83	28.01	0.00			
Residual	39.00	48948.46	1255.09					
Total	40.00	84099.29						
	Coefficients	Standard error	t-Stat	P-value	Lower 95%	Upper 95%	Lower 95.0%	Upper 95.0%
Intercept	650.48	12.11	53.72	0.00	625.99	674.97	625.99	674.97
20.00	–0.91	0.17	–5.29	0.00	–1.25	–0.56	–1.25	–0.56

While the maximum elongation was observed in non-heat-treated samples, the elongation values significantly decreased in the samples subjected to aging, fig. 6. It has been estimated that lower internal stresses will occur with quenching at higher temperatures. To support this view, the average elongation values of 15 samples were determined as 5.7% for

100 °C and 5.3% for 20 °C [13]. On the other hand, Mahathaninwong *et al.* [12] had reached as high as 12% elongation when samples were quenched in room temperature, however, the UTS was significantly low in their study.

According to the ANOVA analysis, tab. 6, eq. (3) suggests that the minimum elongation at fracture value of this alloy is around 5.06% which has the tendency to increase with increased quenching temperature.

$$y = 0.0057x + 5.0658R^2 = 1 \quad (3)$$

where y is the elongation and x – the quenching temperature.

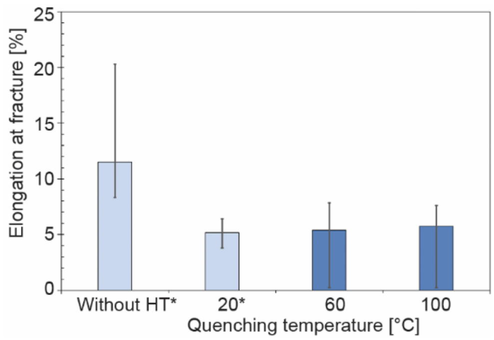

Figure 6. Elongation values obtained at different quenching temperatures [13]

Table 6. The ANOVA test results comparing the relationship between elongation and quenching temperature

	df	SS	MS	F	Significance F			
Regression	1.00	1.37	1.37	0.41	0.52			
Residual	39.00	129.65	3.32					
Total	40.00	131.02						
	Coefficients	Standard error	t-Stat	P-value	Lower 95%	Upper 95%	Lower 95.0%	Upper 95.0%
Intercept	5.07	0.62	8.13	0.00	3.81	6.33	3.81	6.33
20.00	0.01	0.01	0.64	0.52	–0.01	0.02	–0.01	0.02

Based on the works of Caceres [15], Tiryakioglu and Campbell [16], quality index (QT) of the tensile properties were calculated. The results are summarized in fig. 7. It can be seen that as the quenching temperature is increased, QT value has a trend to decrease. It is important to note that the scatter also increases with increase quenching temperature.

Similar to test results in fig. 7, according to the ANOVA analysis, tab. 7, eq. (4) shows that the quality index of this alloy has the tendency to decrease with increased quenching temperature and also a potential of highest 758 quality index value. Based on Mahathaninwong *et al.* [12] results, the quality index in that study corresponds to 532.

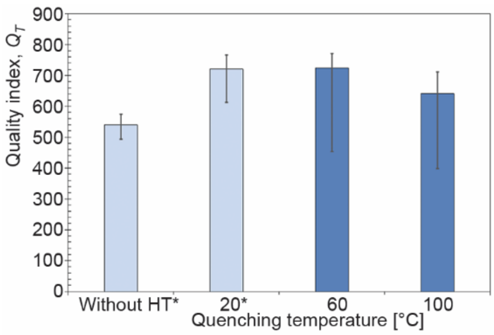

Figure 7. Quality index obtained at different quenching temperatures [13]

$$y = -1.0321x + 758.27R^2 = 1 \quad (4)$$

where y is the quality index and x – the quenching temperature.

Table 7. The ANOVA test results comparing the relationship between quality index and quenching temperature

	df	SS	MS	F	Significance F			
Regression	1.00	45646.06	45646.06	8.22	0.01			
Residual	39.00	216455.53	5550.14					
Total	40.00	262101.59						
	Coefficients	Standard error	t-Stat	P-value	Lower 95%	Upper 95%	Lower 95.0%	Upper 95.0%
Intercept	758.27	25.46	29.78	0.00	706.77	809.77	706.77	809.77
20.00	–1.03	0.36	–2.87	0.01	–1.76	–0.30	–1.76	–0.30

The SEM analyses were performed on the fracture surfaces of tensile test specimens. SEM images of two samples quenched at 60 °C and 100 °C with much lower than expected tensile strength and elongation are shown in fig. 8. An intergranular fractured surface morphology like hot tearing is seen in these samples. Spheroidal grains are clearly visible on the fracture surface. It is thought that the molten secondary phase has formed a continuous brittle area along the grain boundaries perpendicular to the axis of the sample and a crack, triggered by a possible bifilm, causing the sample to break at a much lower than the average stress.

On the fractured surface, a ductile structure, which is mostly spongiform in appearance, has been observed. There are some straight line-like porosities, which are seen in similar studies [11, 17-21]. It is thought that the source of these faults is double oxide layers (bifilms) that was incorporated into the material during the production of the alloy. The appearance of a large number of heterogeneously distributed crack-like faults, shown in a sample with such ductile behavior, demonstrates the presence of bifilms [22, 23].

Figure 9 shows SEM images of a sample quenched at 100 °C which was prematurely fractured. It is observed that due to the poor machining quality of the tensile specimen, a notch effect had facilitated the fracture, fig. 9(a), and several bifilm-induced porosities are also seen in this sample, fig. 9(b).

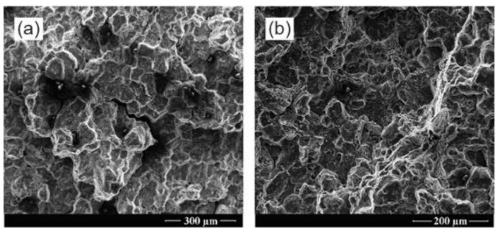

Figure 8. The SEM images of two samples quenched at: (a) 60 °C and (b) 100 °C with low mechanical properties

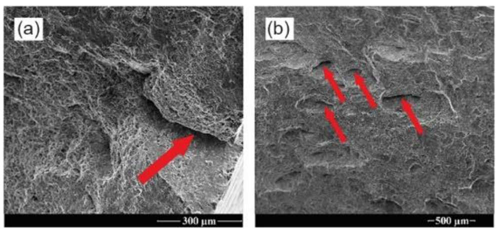

Figure 9. The SEM images of a sample quenched at 100 °C with low mechanical properties

Conclusion

Semisolid processing is a manufacturing technique that can be used to fabricate complex aluminum shapes at low cost with fewer steps, and high quality. Homogeneous spherical grain structure should be obtained in order to perform semisolid forming processes of Al alloys. In this study, the desired spherical grain structure was obtained by SIMA process and T6 heat treatment was applied to the 7075 alloy after SIMA process with different parameters to investigate the effects of quenching temperature, solutionizing and artificial aging times on microstructural and mechanical properties.

In this work, it was found that:

- The secondary phases precipitated in SIMA 7075 alloy dissolve homogeneously when kept at 490 °C after 45 minutes.
- The highest yield and ultimate tensile strength were obtained in samples quenched at 60 °C.
- At 100 °C quenching temperature, the dissolved secondary phases precipitated as coarse particles, especially at grain boundaries.
- The average tensile strengths were 627 and 545 MPa for quenching in water at 60-100 °C respectively. The average yield strengths were 576 and 461 MPa for quenching in water at 60-100 °C respectively. At two different quenching temperatures, no significant change in average elongation values were observed which was around 5.5%.
- The optimum aging time at 120 °C was determined to be 48 hours from the hardness tests.
- Bifilms, porosities and low machining quality have a role in reducing the tensile strength and elongation.
- Formation of continuous brittle secondary phases surrounding the solid spheroidal particles perpendicular to the axis of the sample may lead to intergranular fracture at a low stress.
- Based on ANOVA tests, the correlation between mechanical properties, y, and quenching temperature, x, was found to be:
  - yield strength: $y = -1.3408x + 614.23R^2 = 1$
  - ultimate tensile strength: $y = -0.9057x + 650.48R^2 = 1$
  - elongation at fracture: $y = 0.0057x + 5.0658R^2 = 1$
  - quality index: $y = -1.0321x + 758.27R^2 = 1$

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References

[1] Spencer, D. B., *et al.*, Rheological Behavior of Sn-15 Pct Pb in the Crystallization Range, *Metallurgical and Materials Transactions B*, *3* (1972), July, pp. 1925-1932
[2] Flemings, M. C., *et al.*, Rheocasting, *Materials Science and Engineering, 25* (1976), Sept.-Oct., pp. 103-117
[3] Hirt, G., Kopp. R., *Thixoforming: Semi-Solid Metal Processing*, Wiley-VCH, Weinheim, Germany, 2009
[4] Atkinson, H. V., *et al.*, Recrystallisation in the Semi-Solid State in 7075 Aluminium Alloy, *Materials Science and Engineering A*, *490* (2008), 1-2, pp. 266-276
[5] Sirong, Y., *et al.*, Microstructure Evolution of SIMA Processed Al2024, *Materials Science and Engineering A, 420* (2006), 1-2, pp. 165-170
[6] Jiang, J., *et al.*, Comparison of Microstructural Evolution of 7075 Aluminum Alloy Fabricated by SIMA and RAP, *Journal of Materials Processing Technology*, *238* (2016), Dec., pp. 361-372
[7] Binesh, B., Aghaie-Khafri, M., Microstructure and Texture Characterization of 7075 Al alloy during the SIMA Process, *Materials Characterization*, *106* (2015), Aug., pp. 390-403

[8] Das, P., Dutta, P., Globularization of Primary Phase of Al-7Si-0.3Mg Alloy during Cooling Slope Processing and Isothermal Holding, *Transactions of the Indian Institute of Metals*, *74* (2021), Feb., pp. 1241-1251
[9] Cai, S. W., *et al*., Study on the Strengthening Mechanism of Two-Stage Double-Peaks Aging in 7075 Aluminum Alloy, *Transactions of the Indian Institute of Metals*, *73* (2020), Nov., pp. 109-117
[10] Dong, X., *et al.*, Microstructure and Microhardness of Hot Extruded 7075 Aluminum Alloy Micro-Gear, *Journal of Materials Processing Technology*, *219* (2015), May, pp. 199-208
[11] Tan, E., *et al.*, The Effect of Melt Quality and Quenching Temperature on the Weibull Distribution of Tensile Properties in Aluminium Alloys, *Materialwissenschaft und Werkstofftechnik*, *46* (2015), 10, pp. 1005-1013
[12] Mahathaninwong, N., *et al.*, T6 Heat Treatment of Rheocasting 7075 Al Alloy, *Materials Science and Engineering A*, *532* (2012), Jan., pp. 91-99
[13] Guner, A. T., *et al.*, Microstructural and Mechanical Evolution of Semisolid 7075 Al Alloy Produced by SIMA Process at Various Heat Treatment Parameters, *Arabian Journal for Science and Engineering*, *44* (2019), Aug., pp. 1243-1253
[14] Tan, E., *et al.*, Improvement in Metallurgical Properties of Gravity Die Cast 2024-T6 Aluminum Alloy via Cryogenic Process, *TMS 148th Annual Meeting, Shape Casting: 7th Int. Symp. Celebrating Prof. John Campbell's 80th Birthday*, (ed. M. Tiryakioglu, W. Griffiths, M. Jolly), 2019, Springer Nature, Switzerland, pp. 263-271
[15] Caceres, C. H., A Rationale for the Quality Index of Al-Si-Mg Casting Alloys, *International Journal of Cast Metals Research*, *12* (2000), 6, pp. 293-299
[16] Tiryakioglu, M., Campbell, J., Quality Index for Aluminum Alloy Castings, *International Journal of Metalcasting*, *8* (2014), 3, pp. 39-42
[17] Davies, D. P., Jenkins, S. L., Assessment of a Controlled Solidification Aluminium Investment Casting Technique for Use in Helicopter Gearboxes, *Materials Science and Engineering A*, *651* (2016), Jan., pp. 449-460
[18] Tiryakioglu, M., *et al.,* Evaluating Structural Integrity of Cast Al-7% Si-Mg Alloys via Work Hardening Characteristics: 1. Concept of Target Properties, *Materials Science and Engineering A*, *368* (2004), 1-2, pp. 205-211
[19] Dorum, C., *et al*., Numerical Modelling of Magnesium Die-Castings using Stochastic Fracture Parameters, *Engineering Fracture Mechanics*, *76* (2009), 14, pp. 2232-2248
[20] Dispinar, D., *et al*., Degassing, Hydrogen and Porosity Phenomena in A356, *Materials Science and Engineering A*, *527* (2010), 16-17, pp. 3719-3725
[21] Dispinar, D., Campbell, J., Porosity, Hydrogen and Bifilm Content in Al Alloy Castings, *Materials Science and Engineering A*, *528* (2011), 10-11, pp. 3860-3865
[22] Uludag, M., *et al.*, Relationship between Machinability, Microstructure, and Mechanical Properties of Al-7Si Alloy, *Journal of Testing and Evaluation*, *46* (2018), 6, pp. 2592-2603
[23] Yuksel, C., *et al.*, Quality Evaluation of Remelted A356 Scraps, *Arch Foundry Eng, 16* (2016), 3, pp. 151-156

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