Materials Science and Engineering A 390 (2005) 3–12

MATERIALS SCIENCE & ENGINEERING A

www.elsevier.com/locate/msea

Thixoforming 7075 aluminium alloys

S. Chayong[1], H.V. Atkinson*, P. Kapranos

Department of Engineering Materials, University of Sheffield, Sir Robert Hadfield Building, Mappin Street, Sheffield S1 3JD, UK

Received 13 October 2003; received in revised form 18 March 2004; accepted 4 May 2004

Abstract

Commercially extruded 7075 alloy (extrusion ratio of 16:1) has been used as a feedstock for thixoforming in order to investigate thixoformability of a high performance aluminium alloy. The microstructure in the semi-solid state consists of fine spheroidal solid grains surrounded by liquid. The results of thixoforming with one step, two-step and three-step induction heating regimes are presented. Typical defects in poorly thixoformed material (e.g. liquid segregation, impedance of flow by unrecrystallised grains and porosity) are shown alongside successfully thixoformed material (thixoforming temperature of between 615 and 618 °C with a three-step induction heating regime). The highest yield strength and elongation obtained for material thixoformed into a simple graphite die and heat treated to the T6 condition is 478 MPa and 6.9% elongation. For thixoforming at 615 °C into a tool steel die heated to 250 °C, the highest yield strength and elongation obtained are 474 MPa and 4.7% (ram velocity 2000 mm/s). These values (particularly for strength) are approaching those of 7075 in the wrought heat treated condition (505 MPa and 11% elongation).

Keywords: Thixoforming processing; 7075 Aluminium alloy; Microstructures; Properties

1. Introduction

Thixoforming is a semi-solid metal (SSM) processing route. The process has been established as a relatively new technology for metal forming [1]. For the alloy to be shaped in the semi-solid state it must have a non-dendritic structure. It then behaves as a 'thixotropic' slurry in which viscosity decreases with increasing shear rate and at constant shear rate the viscosity decreases with increasing time. Such alloy slurries flow in a laminar manner, which allows for uniform die filling as opposed to the turbulent flow associated with the fully liquid state (casting) forming processes. Furthermore, the process has high capability for near net shaping because there is less solidification shrinkage than for fully liquid casting. Components have high mechanical integrity with little porosity, making the replacement of heavier materials such as steel for safety critical components technologically feasible. In addition, sections can be made thinner and hence lighter. The main disadvantage of thixoforming is the cost premium for the starting material, which must be in such a condition that when it is reheated into the semi-solid state the required microstructure is obtained. For commercial thixoforming, magnetohydrodynamic (MHD) stirring is usually used to produce the starting material. Various other routes to creating the starting material are available including recrystallisation and partial melting (RAP) [2]. In this route, material is warm worked, below the recrystallisation temperature. When it is then reheated and recrystallisation occurs, liquid penetrates the recrystallised boundaries to give spheroids surrounded by liquid. It is this route which is utilised in the work described here.

In thixoforming, a cylindrical slug of the appropriate size is cut from a bar of starting material (in this case in the extruded state). The slug is then reheated (usually with induction heating) into the semi-solid condition with approximately 30–50% fraction liquid. The temperature of the slug must be carefully controlled in order to obtain a homogeneous (in the sense of uniform volume fraction liquid and uniform spheroid size) microstructure prior to forming. Finally, the slug is forced into the die. Thixoforming is in commercial use but only with the casting alloys such as A356 and A357. These alloys give strength between 220–260 MPa and 8–13% elongation [3]. Therefore one of the major challenges

* Corresponding author. Present address: Department of Engineering, University of Leicester, University Road, Leicester LE1 7RH, UK. Tel.: +44 0116 2231019; fax: +44 0116 2522619.

E-mail address: hva2@le.ac.uk (H.V. Atkinson).

[1] Present address: Faculty of Engineering, University of Ubonratchathani, Warinchamrap, Ubonratchathani 34190, Thailand.

0921-5093/$ – see front matter © 2004 Elsevier B.V. All rights reserved.
doi:10.1016/j.msea.2004.05.004

Table 1
Thixoforming experiments

Experiment	Die material/die volume	Thixoforming temperature (°C)	Ram speed (mm/s)	Heating conditions
(1) Single-step heating	Cross-shaped graphite/117000 mm^3	615, 618, 620	500	Rapid heating to 620 °C with total heating time of ∼3 min
(2) Three-step heating (a)	Cross-shaped graphite/117000 mm^3	616	500	Rapid heating to 500 °C (1 min hold), then slower rate to 575 °C (1 min hold) and 616 °C with total heating time of ∼7 min
(3) Three-step heating (b)	Cross-shaped graphite/117000 mm^3	616	750	Rapid heating to 500 °C, then slower rate to 575 °C and 616 °C with total heating time of ∼7 min
(4) Three-step heating (c)	Cross-shaped graphite/117000 mm^3	616	1000	Rapid heating to 500 °C, then slower rate to 575 °C and 616 °C with total heating time of ∼7 min
(5) Three-step heating (d)	Plate-shaped tool steel heated to 250 °C/149500 mm^3	614	1000	Rapid heating to 500 °C, then slower rate to 575 °C and 616 °C with total heating time of ∼7 min

is to develop thixoforming for the higher performance alloys which are normally wrought e.g. 2000 series, 6000 series and 7000 series. The difficulties in thixoforming these alloys centre around the wide interval over which solidification occurs, which can lead to hot tearing [4], and the steep slope for the fraction liquid versus temperature curve in the region of 40% liquid, which leads to narrow processing windows [5]. For example, for 6061 the temperature window for processing between 30 and 50% liquid is only a few K [6].

This study is focused on high strength 7075 wrought aluminium alloy. It is typically used for aerospace applications and is heat treatable to obtain a yield strength of 505 MPa and 11% elongation [7]. The precipitation hardening phase is $MgZn_2$, provided the ageing temperature is below 200 °C [8]. 7075, with more than 1% Cu also precipitates $CuMgAl_2$ [8]. The hardening precipitates are up to 0.01 μm in size. Dispersoid particles are also present, based on the transition elements Cr, Zr and Mn. These include $Al_{12}Mg_2Cr$ [9] and $Al_{18}Mg_3Cr_2$ (E-phase) [10]. These range in size from 0.5–2 μm and play an important role in grain and subgrain boundary pinning. The E-phase particles are of sufficient size and volume fraction to make the alloy difficult to recrystallise. Fe and Si are present in the alloy as impurities. They give rise to constituent phase particles, which are detrimental to most of the mechanical properties of the alloy [9] and are resistant to dissolution. They range in size from 1 to 30 μm.

7075 Alloy has been shown to have the potential for thixoforming [11–14]. Here results are presented on:

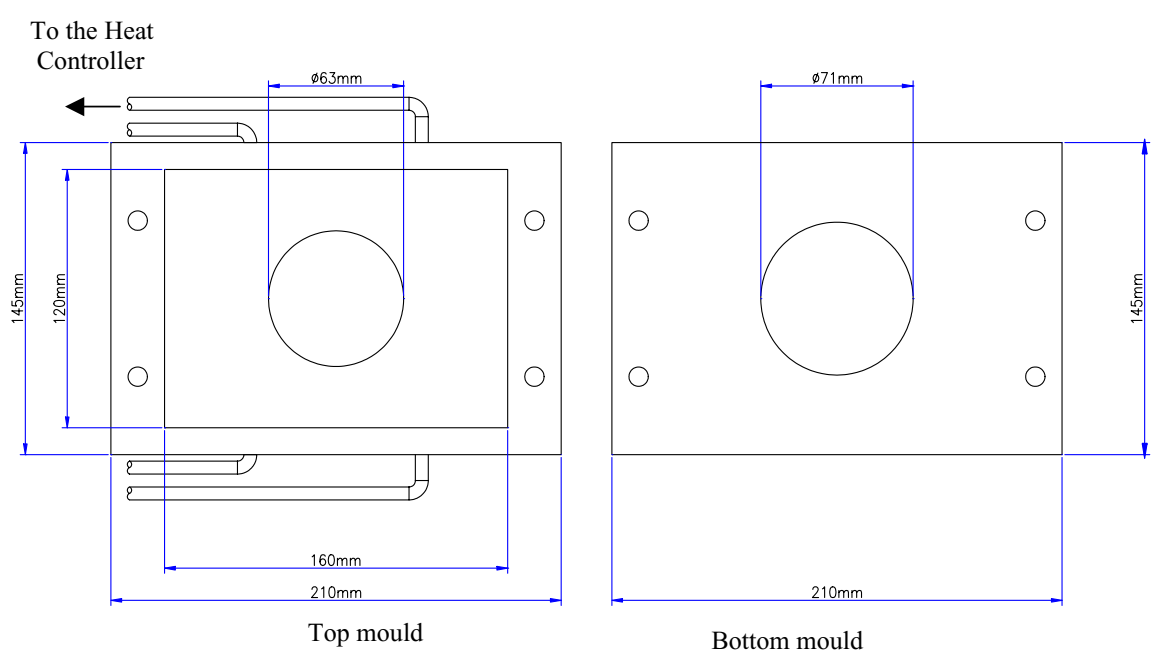


Fig. 1. Schematic illustrates the top view of the tool steel die (plate-shape).

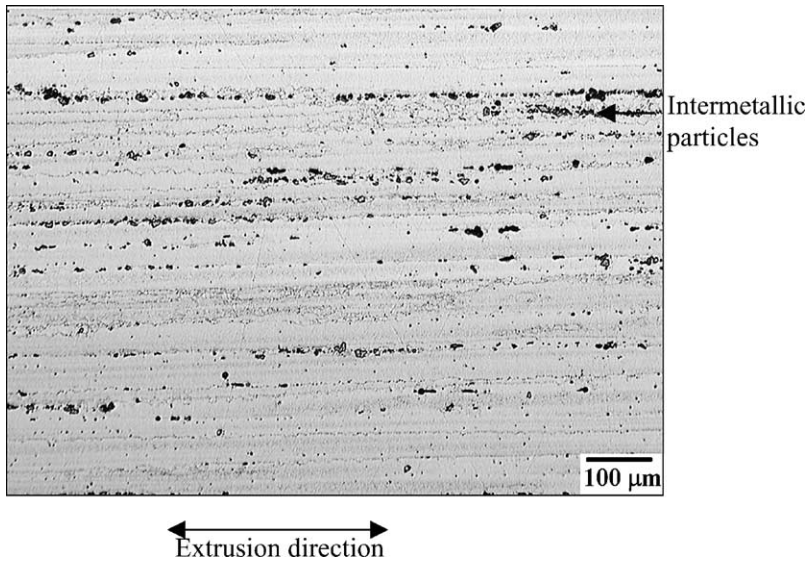


Fig. 2. As extruded microstructure of the wrought 7075 alloy. Intermetallic particles appear as stringers aligned with the extrusion direction.

microstructural evolution in the semi-solid state; thixoforming with different die materials and ram velocities; mechanical properties of thixoformed material in the T6 condition.

2. Experimental

The material used was a commercially wrought 7075 aluminium alloy supplied by Severn Metals Ltd. as 64 mm bar

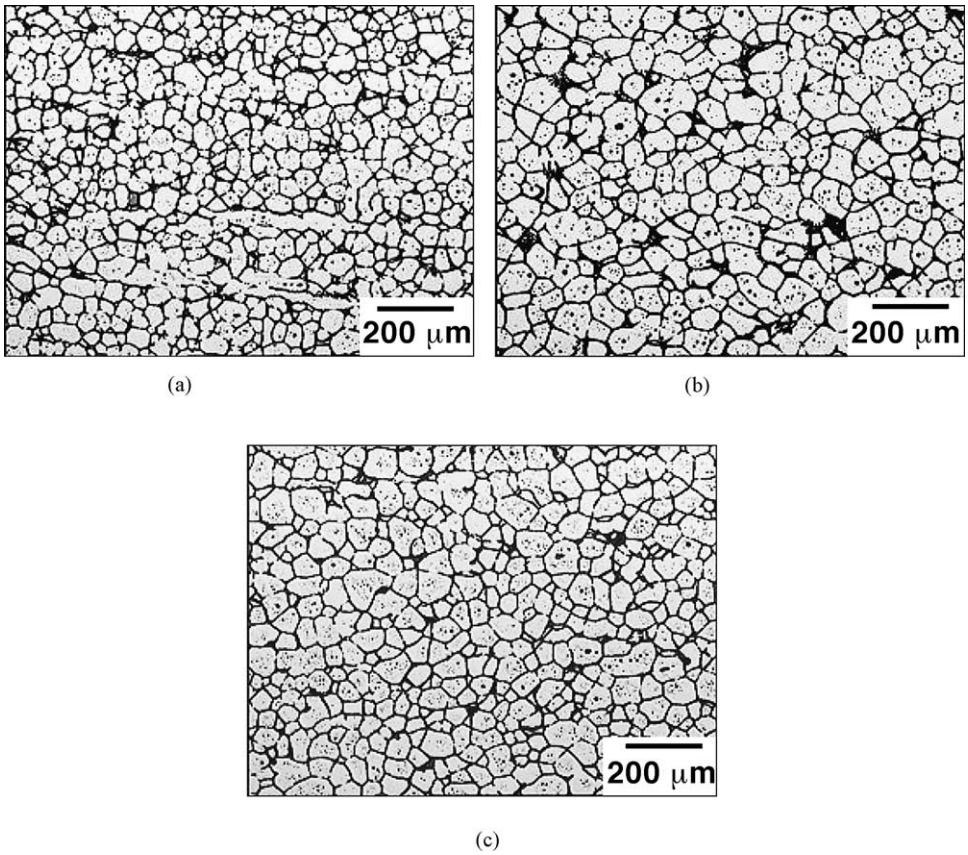


Fig. 3. Microstructural changes after induction heating to semi-solid temperature and hold time (in parenthesis), (Barker's etch): (a) 475 °C (2 min), 620 °C (1 min); (b) 475 °C (1 min), 575 °C (2 min), 620 °C (2 min); (c) 475 °C (1 min), 575 °C (1 min), 620 °C (20 s).

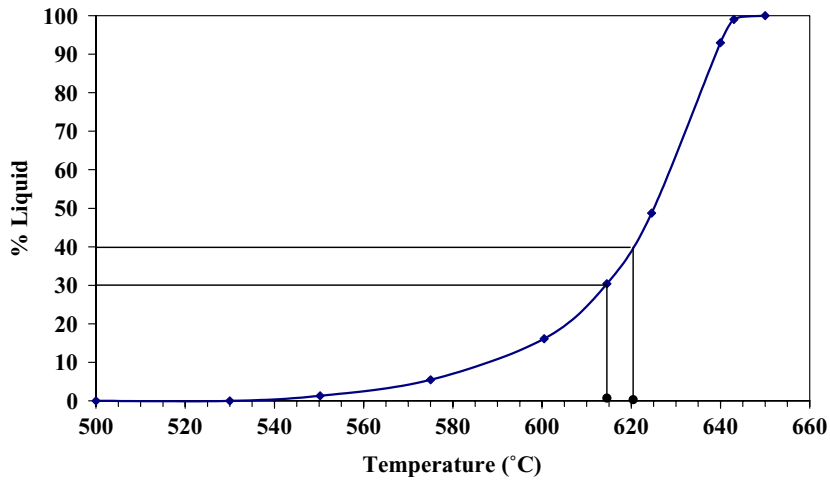


Fig. 4. Percent liquid against temperature of 7075 alloy obtained from DSC analysis at a constant heating rate of 18 °C/min using the Dupont 910 apparatus.

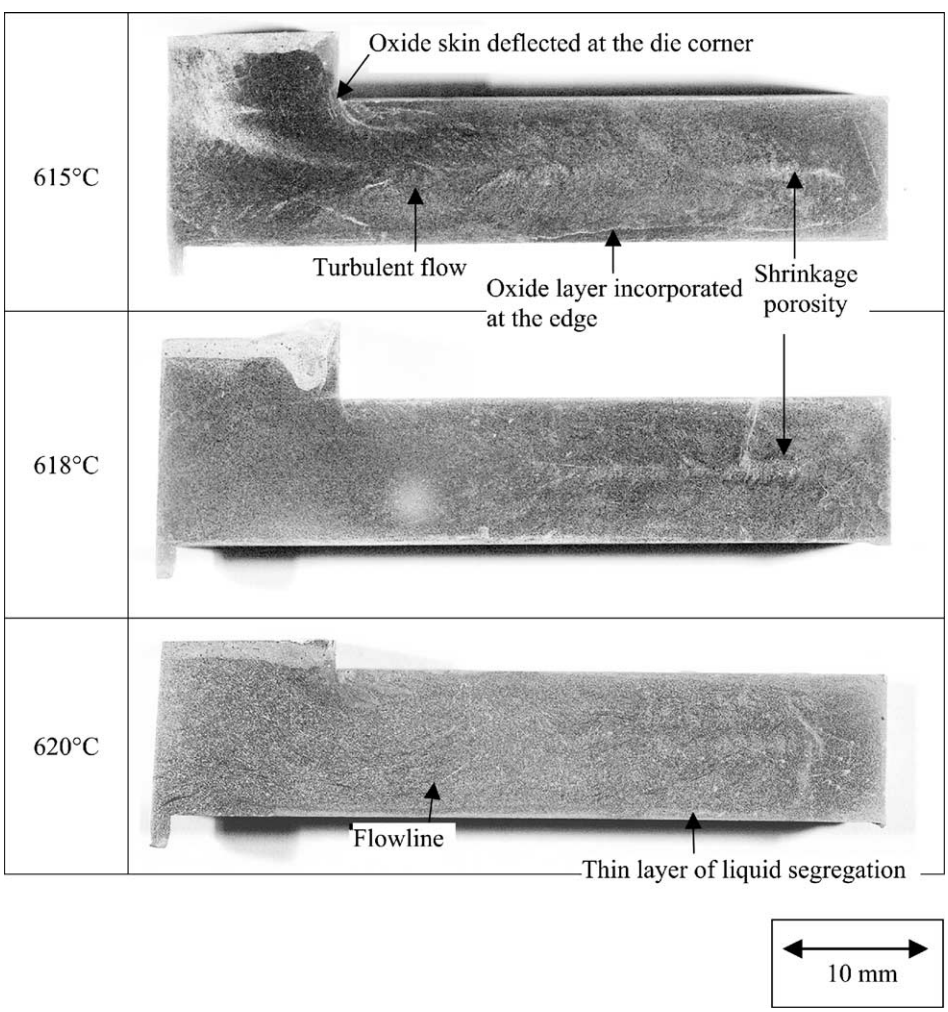


Fig. 5. Low magnification images of sections through the selected fingers along the flow direction of the finger. All fingers are thixoformed using single-step heating schedule and 500 mm/s ram velocity in graphite dies (Macroetching).

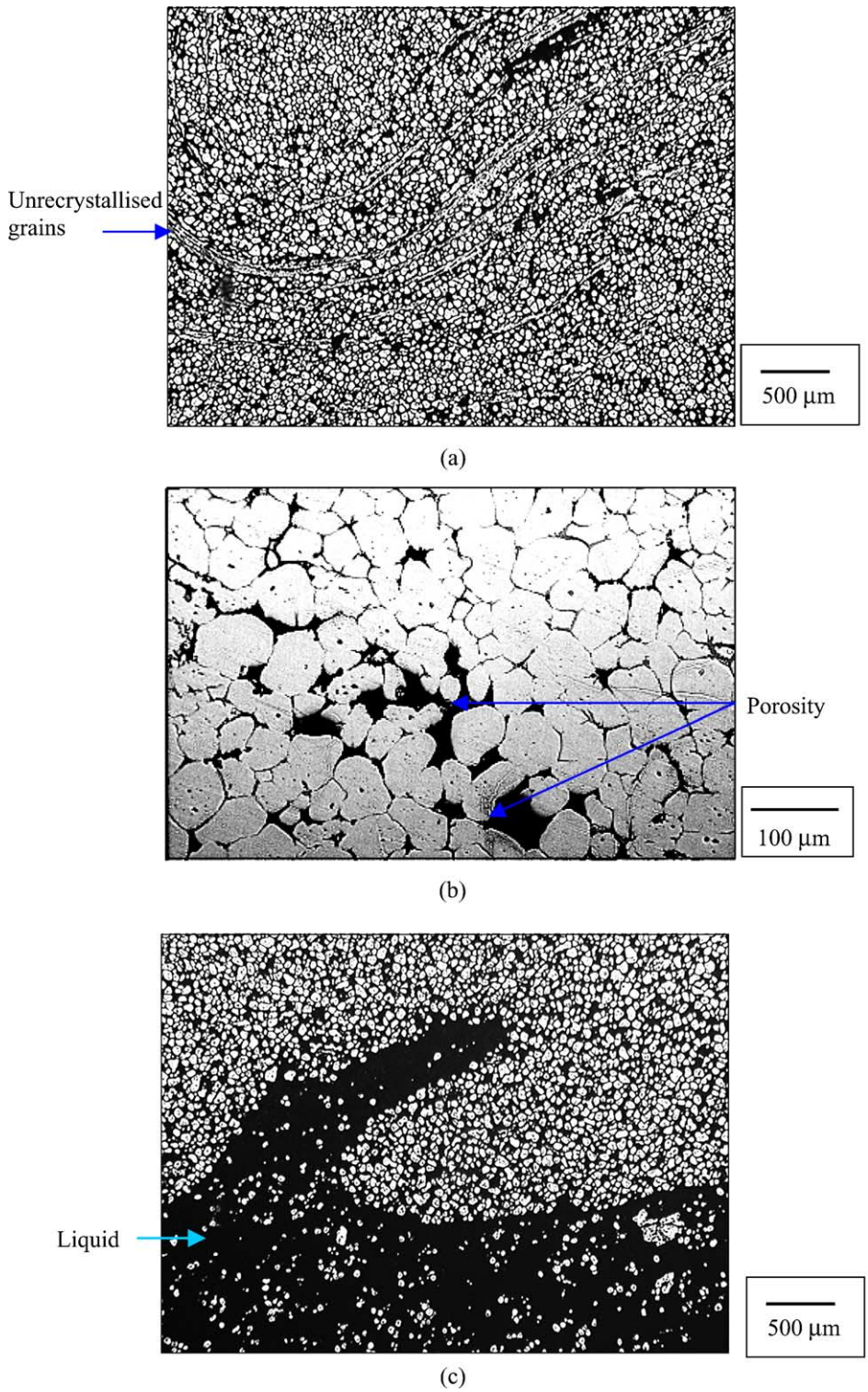


Fig. 6. Microstructures illustrating (a) the unrecrystallised grains showing flowlines and (b) porosity found at the central line through the finger thixoformed by single-step heating at 615 °C. The microstructure in (c) shows turbulent flow caused by excessive liquid found in the finger thixoformed by single-step heating at 620 °C; 40% liquid (Tuckers'etch).

with composition of Al-5.3Zn-2.34Mg-1.51Cu-0.22Cr-0.33 Fe-0.1Si-0.07Ti (numbers indicate wt.%). The alloy has undergone extrusion with a ratio of 16:1 and T6511 treatment (i.e. T6 followed by stress-relief by stretching, followed by minor straightening, in order to comply with standard tolerances and eliminate the distortion caused by quenching).

The liquid fraction against temperature curve was estimated by differential scanning calorimetry (DSC) using

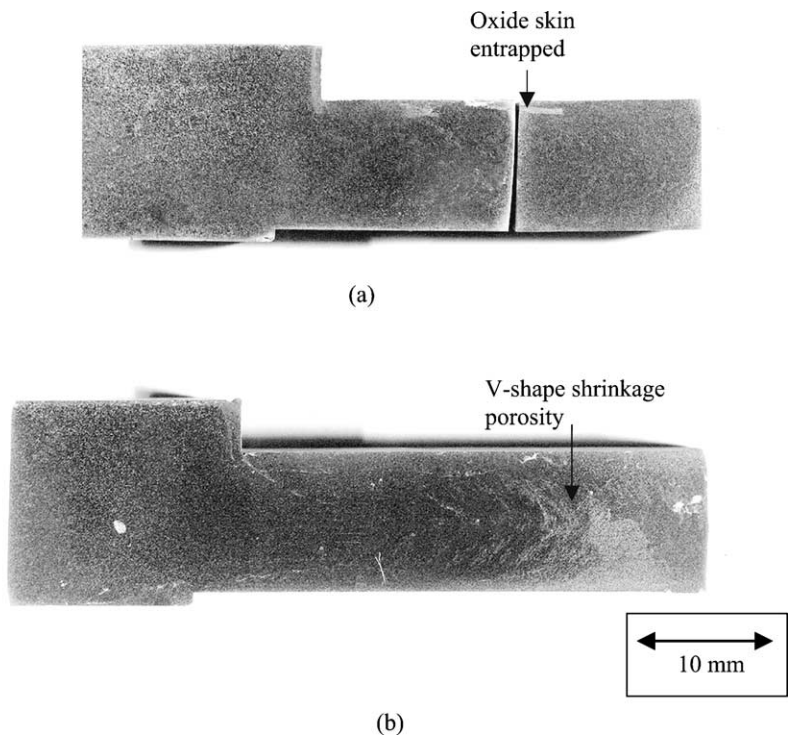


Fig. 7. Low magnification images of sections through the selected fingers along the flow direction of the finger. All fingers are thixoformed using graphite dies (Macroetching): (a) three-step heating; 575 °C (1.5 min), 585 °C (1.5 min) and finally thixoformed at 617 °C. Ram velocity = 750 mm/s. Total heating time = 7 min 14 s; (b) three-step heating; 500 °C (1.5 min), 575 °C (1.5 min) and finally thixoformed at 615 °C. Ram velocity is 1000 mm/s. Total heating time = 6 min.

a Dupont 910 DSC system. The results show that the semi-solid region of 30–50% liquid content is between 615 and 625 °C. Microstructural change in the semi-solid state was investigated with a small induction heating rig (see [15] for more details) with small cylinders of 12 mm in diameter and 16 mm in height machined from the as-received material.

Thixoforming was carried out on the vertically upwards acting thixoforming press at Sheffield University [16]. 7075 starting material for thixoforming was 60 mm in diameter and 64 mm in height. One-step, two-step and three-step induction heating regimes as described in Table 1 were tested. The temperature of the slug was monitored by a K-type thermocouple embedded in the slug. After the required temperature was reached, the thermocouple was removed and the heated slug raised and compressed into the die at a constant ram speed. The first die was made of graphite with a cross-shaped cavity with four ‘fingers’ (as shown schematically in [4]). Fig. 1 shows the second die made of tool steel heated to 250 °C with a plate-shaped cavity. The experimental details for thixoforming are summarised in Table 1. The effects of varying ram velocity are studied in tests Nos. 2–4. The effect of die temperature and die material is studied in test No. 5 using the plate-shaped tool steel die heated to 250 °C and coated with carbon solution.

Eight tensile samples (gauge length 25 mm, gauge diameter 4 mm, total length 50 mm) were machined from each thixoformed product (two from each finger as shown in [4]) and T6 heat treated. The solution treatment was at 465 °C for 16 h, followed by quenching then ageing at 125 °C for 24 h. The solution time is longer than the wrought standard (usually 1–2 h) in order to dissolve non-equlibrium and low melting point eutectics and reduce the segregation occurring during thixoforming.

Microstructural characterisation was carried out on the as-received materials, induction-heated and thixoformed materials. Specimens were mechanically polished and electrolytically etched with Barker’s reagent (5 ml HBF_4 (48%), 95 ml H_2O) by immersion for 90 s at 20 V. The microstructures were examined using an optical microscope under gypsum-polarised light. Tucker’s etch was also used (HCl 45 ml, HNO_3 15 ml, HF 15 ml, H_2O 25 ml). The etchant was diluted with 50% water to slow down the reaction.

3. Results and discussion

3.1. Microstructures before and after RAP process

The initial as-received microstructure of the wrought 7075 is shown in Fig. 2, and consists of elongated grains with stringers of intermetallic particles (these are identified elsewhere [15]). In Fig. 3, examples of microstructures

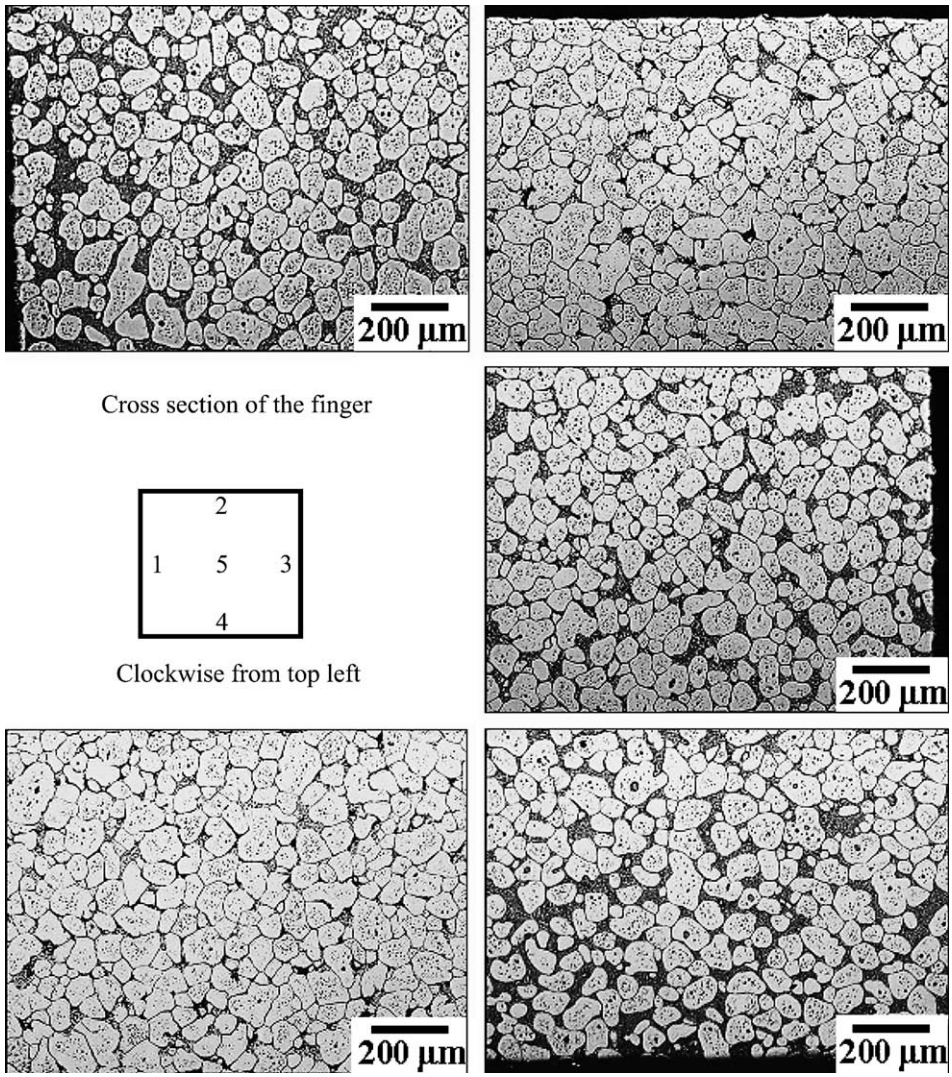


Fig. 8. Optical micrographs show microstructural distribution across the section of the thixoformed 7075 using three-step heating. Ram velocity = 750 mm/s, total heating time = 7 min 14 s.

after induction heating tests in the small rig are shown. Recrystallisation has occurred with liquid penetration of the boundaries, but in Fig. 3a, for example, there are some unrecrystallised grains. Three-step heating (Fig. 3b and c) gives fully recrystallised structures. If the hold at 620 °C is brief, microstructural coarsening is inhibited (Fig. 3c). The apparent quantity of liquid is less than that present at temperature because the quench rate is

Table 2
Mechanical properties of thixoformed sample in the as-thixoformed, thixoformed by single step heating and T6 treatment, thixoformed by three-step heating and T6 treatment compared with the 7075 T6 standard (ASM)

Experiment/condition	Average yield strength (MPa)	Average elongation (%)
As-thixoformed, experiment 1 (graphite die)	225.3 ± 27	2.0 ± 1.4
As-thixoformed, experiment 5 (heated tool steel die)	235 ± 1.8	2.7 ± 0.6
Single-step heating thixoformed (experiment 1) + T6	435.9 ± 8.1	2.5 ± 0.5
Three-step heating thixoformed (experiment 2) + T6	477.6 ± 4.7	3.0 ± 0.5
Three-step heating thixoformed (experiment 3) + T6	470.0 ± 2.3	3.4 ± 1.1
Three-step heating thixoformed (experiment 4) + T6	472.0 ± 4.5	2.4 ± 0.3
Three-step heating thixoformed (experiment 5, Heated tool steel die) +T6	467.0 ± 5.7	3.2 ± 0.8
7075 T6-standard	505	11

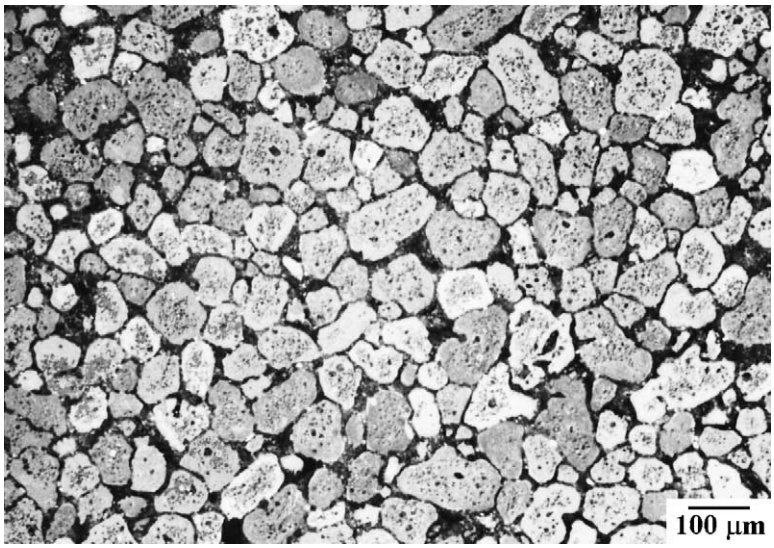


Fig. 9. Typical micrograph viewed under polarised light showing the microstructure parallel to flow direction of the wrought 7075 thixoformed in a heated tool steel die at 614 °C. Total heating time is 8 min. 5 s. ram speed = 1000 mm/s (Barker's etch).

insufficient to prevent some solidification onto the spheroids during cooling [17]. Fig. 4 shows the liquid fraction versus temperature curve for 7075 from the DSC test. The semi-solid range of 30–40% liquid is between 614 and 622 °C. More detailed study of the effects of the one-step, two-step and three-step induction heating regimes [15] suggests that the particles, such as E-phase, which effectively pin some of the boundaries and prevent full recrystallisation are coarsened and dissolved by the three-step regime.

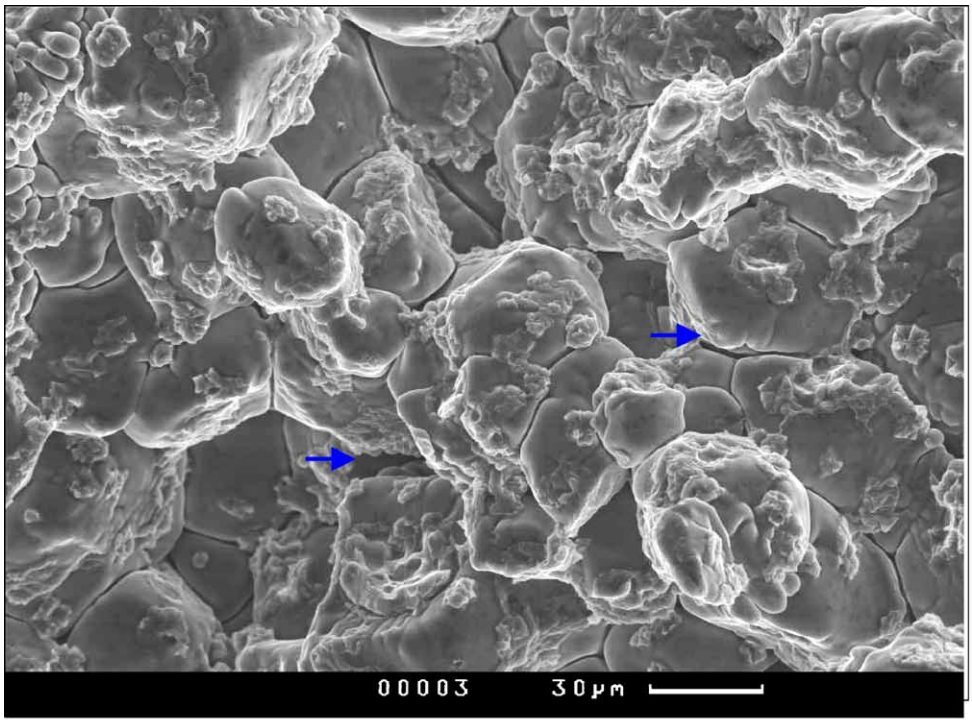
3.2. Thixoforming

Fig. 5 shows low magnification images of sections through fingers thixoformed using one-step heating schedules with varying thixoforming temperatures and constant ram velocity of 500 mm/s. Typical defects are labelled. Oxide skin may be deflected by the sharp corner at the entrance of the die and incorporated into the essentially laminar flow. The sharp corner can also cause some turbulence, which is undesirable in thixoforming [18]. Shrinkage porosity tends to occur on the centreline near the ends of the fingers and at the lower thixoforming temperatures, suggesting that the cooling rate is too fast for a material with a relatively wide temperature range over which the final solidification is occurring (see Fig. 4). The oxide incorporation could be avoided with oxide stripping devices, which are used by some of the commercial manufacturers to skim the oxide off the surface of the billet before it enters the die. Fig. 6 shows defects in more detail with unrecrystallised grains potentially obstructing the flow of slurry in Fig. 6a, centre-line shrinkage porosity in Fig. 6b and liquid/solid segregation with turbulent flow taken from the central edge of the finger in Fig. 6c. The excessive liquid generated at above 620 °C may cause the turbulence by the sharp corner on the die.

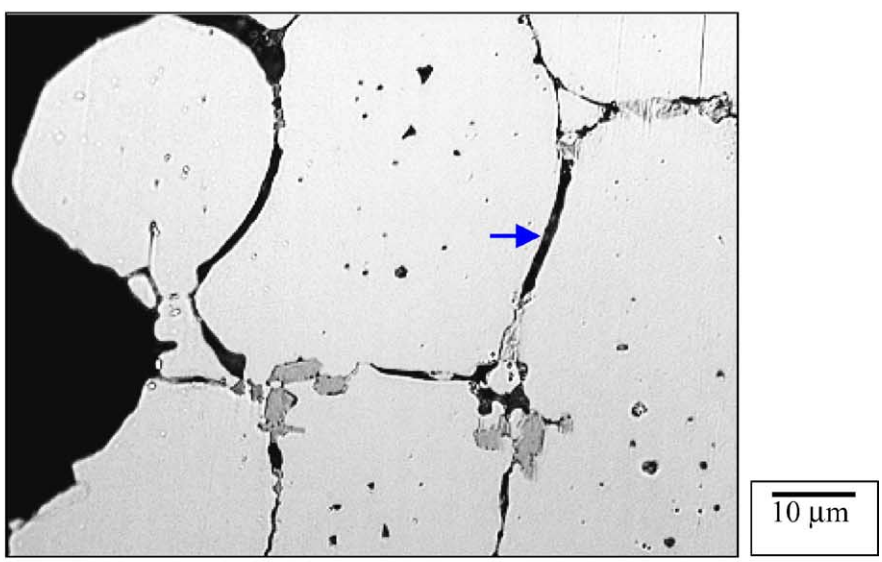
The thixoforming temperature for 7075 should thus not be higher than 620 °C to prevent liquid segregation. In addition, for stability of slugs, the temperature used in subsequent thixoforming experiments was between 615 and 618 °C. An intermediate ram velocity of 750 mm/s tends to give more successful thixoforming than a higher velocity of 1000 mm/s (Fig. 7). Examination of the microstructure through the finger for the higher velocity shows liquid segregation. At 750 mm/s, there is a relatively uniform semi-solid structure across the cross-section (Fig. 8). Three-step heating in general gives more uniform thixoformed microstructures with fewer defects than the one step heating.

The tool steel die was successfully filled with the conditions shown in Table 1. The microstructure of thixoformed 7075 in Fig. 9 exhibited equiaxed solid grains. The liquid phase was found at the boundaries and the triple points. Relatively small liquid pools were also located inside the grains. Defects found here include solidification shrinkage due to premature freezing especially at the end of the plates. However, the density tests shows that density of thixoformed product improved with use of the tool steel heated die.

The average yield strengths of thixoforming samples are summarised in Table 2. The yield strength (0.2% off set) of the as-thixoformed products (without heat treatment) is approximately 225–235 MPa. For single step heating with the graphite die, and in the T6 condition, (with the conditions given in Section 2), the average strength was 435 MPa. This increased further with three-step heating in experiment 2 (see Table 1) to 477 MPa. The average for the heated tool steel die was very similar at 467 MPa. For other high performance thixoformed alloys, it has been found that longer solution treatment times than standard are required [19]. The strength attained in wrought alloys may not be fully attainable in thixoformed material, even after heat treatment, because of the contribution to the strength from the



(a) SEI image of the fracture surface



(b) The area near the fracture surface (No etching)

Fig. 10. SEI showing the fracture surface and optical micrograph of the as-thixoformed specimen that has a low value of strength and elongation (UTS = 189.5 MPa, 1% elongation). The arrows indicate the voids along grain boundaries: (a) SEI image of the fracture surface, and (b) the area near the fracture surface.

dislocation structure. Although the strength is approaching that of the wrought target, the thixoformed products exhibited low elongations of around 3% or less, both before and after heat treatment. Elongation is primarily governed by defects. Fig. 10 shows the fracture surface, and cross-section through that surface for a thixoformed specimen with a low value of strength and elongation. Failure has occurred around the spheroids, where what would have been liquid is apparently missing. This may well be shrinkage porosity (i.e. the wide freezing range means that solidifying liquid between the spheroids cannot be fed during volume contraction) or fracture actually through the liquid during solidification as a result of solidification stresses. This may be alleviated through further work on die design and materials. The best combination of properties for a particular specimen (as opposed to the average shown in Table 2) was 478 MPa yield

strength and 6.9% elongation. This was obtained for a finger thixoformed in a graphite die at 616 °C by three-step heating with a ram speed of 500 mm/s and a one minute hold at 500 and 575 °C (experiment 2). For the tool steel die at 250 °C, the highest yield strength for a particular specimen was 474 MPa with an elongation of 4.7% obtained with three-step heating with a thixoforming temperature of 615 °C and a ram speed of 1000 mm/s.

The effect of ram speed on die filling was investigated. At each ram velocity, eight tensile specimens were produced. All samples had undergone the same T6 treatment. The results do not show any significant effect of ram velocity on the yield strength but the effect on elongation is more significant. Two samples of 500 mm/s and three samples from 750 mm/s die velocities had failed early and did not show yield points. At a ram velocity of 1000 mm/s, elongation reduced noticeably and only four samples showed yield with the lowest elongation. It appears that ram speeds between 500–750 mm/s are most suitable for thixoforming this particular shape. Filling velocity should be such as to give a smooth fill and sufficient freezing time but without being so high as to cause turbulence.

4. Conclusions

7075 Aluminium alloy in the extruded state (i.e. utilising the RAP route to a thixoformable microstructure) can be thixoformed and successfully fill the die. For one step and two-step heating, and lower thixoforming temperatures, defects occur including turbulence, liquid segregation, centreline porosity and unrecrystallised grains which could be obstructing flow. In addition, there may be some incorporated oxide. Three-step heating and thixoforming temperatures in the range 616–618 °C give improved results. An intermediate ram velocity of 750 mm/s gives fewer defects than 1000 mm/s. A graphite die and a heated tool steel die both give successful die filling although the mechanical properties of the thixoformed products are better with the former. The average yield strength for material in the T6 condition thixoformed at 616 °C by three-step heating (a 1 min hold at 500 and 575 °C) with a ram speed of 500 mm/s in the graphite die is 477 MPa with an elongation of 3.0%. This compares with the target value of 505 MPa and 11% for the wrought material. Attention to the elimination of defects through die design may help to improve the elongation values. The highest result obtained was a yield strength of 478 MPa with an elongation of 6.9%.

Acknowledgements

S Chayong would like to thank the Royal Thai Government for scholarship support and Ubonratchathani University for secondment. The authors would also like to thank Dr Philip Ward, Dr D.H. Kirkwood and Dr S.C. Hogg for helpful discussions.

References

[1] M.C. Flemings, Metall. Trans. A. 22 (1991) 957.
[2] D.H. Kirkwood, P. Kapranos, Met. Mater. 5 (1) (1989) 16.
[3] M.P. Kenney, J.A. Courtois, R.D. Evans, G.M. Farrior, C.P. Kyonka, A.A. Koch, K.P. Young, 'Metals Handbook®', ninth ed., vol. 15, ASM, Metals Park, Ohio, 1988, pp. 327–338.
[4] D. Liu, H.V. Atkinson, P. Kapranos, W. Jirattiticharoean, H. Jones, Mater. Sci. Eng. A361 (2003) 213.
[5] H.V. Atkinson, D. Liu, in: Y. Tsutui, M. Kiuchi, K. Ichikawa (Eds.), in: Proceedings of the Seventh International Conference on Advanced Semi-Solid Processing of Alloys and Composites, Tsukuba, Japan, September 2002, National Institute of Advanced Industrial Science and Technology, Tsukuba, 2002, p. 51.
[6] H.E. Pitts, H.V. Atkinson, in: A.K. Bhasin, J.J. Moore, K.P. Young, S.P. Midson (Eds.), in: Proceedings of the Fifth International Conference on Semi-Solid Processing of Alloys and Composites, Golden, Colorado, 23–25 June 1998, Colorado School of Mines, Golden, Colorado, 1998, p. 97.
[7] Metals Handbook, Desk Ed., ASM, Metals Park, Ohio, 1984.
[8] I.J. Polmear, Light Alloys Metallurgy of the Light Metals, third ed., Arnold, London, 1995.
[9] J.T. Staley, in: L. Arnberg, O. Lohne, E. Nes, N. Ryum (Eds.), in: Procedings of the Third International Confernce on Aluminium Alloys: Their Physical and Mechanical Properties, vol. 3, Norwegian Institute of Technology, SINTEF, Trondheim, Norway, June 1992, p. 107.
[10] A. Raghavan, J.Y. Koo, J.W. Steeds, B.K. Park, Metall. Trans. A. 16 (1985) 1925.
[11] K.J. Burke, PhD Thesis, Semi-Solid Processing of Aluminium 7075, University of Sheffield, UK, 1998.
[12] M. Kiuchi, J. Yanagimoto, S. Sugiyama, in: A.K. Bhasin, J.J. Moore, K.P. Young, S.P. Midson (Eds.), Proceedings of the Fifth International Conference on Semi-Solid Processing of Alloys and Composites, Colorado School of Mines, Golden, Colorado, 23–25 June 1998, p. 589.
[13] G. Tausig, in: G.L. Chiarmetta, M. Rosso (Eds.), Proceedings of the Sixth Interntional Conference on Semi-Solid Processing of Alloys and Composites, Turin, Italy, 27–29 September 2000, Edimet Spa, Brescia, Italy, p. 489.
[14] R.S. Rachmat, H. Takano, N. Ikeya, S. Kamado, Y. Kojima, in: A. Roosz, M. Rettenmayr, D. Watring (Eds.), Proceedings of the Third International Conference on Solidification and Gravity, Miskolc, Hungary, April 1999, Trans Tech Publications, Zurich, Switzerland, 2000, p. 487.
[15] S. Chayong, H.V. Atkinson, P. Kapranos, Multi-step induction heating regimes for thixoforming 7075 aluminium alloy, Materials Science and Technology 20 (2004) 490.
[16] H.V. Atkinson, in: P.J. Gregson, S.J. Harris (Eds.), Proceedings of the International Conference "Aluminium Alloys 2002", Cambridge, 2–5 July 2002, Materials Science Forum, vols. 396–402, Trans Tech Publications, Zurich, Switzerland, p. 131.
[17] E. Tzimas, A. Zavaliangos, Mater. Sci. Eng. A289 (2000) 228.
[18] A.R.A. McLelland, H.V. Atkinson, P.R.G. Anderson, Mater. Sci. Technol. 15 (1999) 939.
[19] D. Liu, H.V. Atkinson, P. Kapranos, H. Jones, J. Mater. Sci. 39 (2004) 99.