Relationship between degassing conditions and tensile properties of AI–20Si–X P/M products

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Results are reported which show the effect of different degassing modes on the properties of the Al-20Si-3Cu-1Mg powder. The paper complements previous papers [1-3] concerning the conventional and modified degassing of the same powder. This research was mainly directed to study the influence of temperature on the tensile properties, ultimate tensile strength, σ_{UTS} , and elongation, ε , of extrudates obtained of Al-20Si-3Cu-1Mg compacts non-degassed, conventionally degassed, and treated by a modified process, namely degassing assisted by flushing with a depurative gas such as argon or nitrogen. The processing of the Al-20Si-3Cu-1Mg P/M powder must include a degassing step which significantly improves the tensile properties, at room and elevated temperatures, of the products of compacted powder with respect to those of the products whose compacts were non-degassed. It is apparent that degassing assisted by flushing with argon or nitrogen gives products with higher tensile properties than those of the products conventionally degassed under optimal conditions of temperature and time and much higher than those of the non-degassed products. The tensile results are in agreement with the theoretical approach to the gas entrapment and evolution of the aluminium powders presented in previous papers.

1. Introduction

It has been shown that the processing of the Al-20Si-3Cu-1Mg P/M alloy must include a degassing step to remove water vapour and hydrogen [1, 2] and that degassing assisted by flushing with a depurative gas such as argon (Ar) or nitrogen (N₂) increases the efficiency of moisture (H₂O) and hydrogen (H₂) evolution in comparison with the conventional mode of degassing [3].

The fundamental aspects of moisture and hydrogen evolution during degassing of a porous billet have been described in a semi-quantitative manner by means of a thermodynamic and kinetic approach [1-3].

One of the aims of the work presented here was to investigate the effect of temperature on the tensile properties, ultimate tensile strength, σ_{UTS} , and elongation, ϵ , of Al-20Si-3Cu-1Mg P/M extrudates obtained of compacts conventionally degassed and of compacts treated by a modified degassing process, namely, degassing assisted by flushing with a depurative gas such as Ar or N2. The tensile properties of extrudates of non-degassed material are used as a reference. This work also aimed to verify experimentally the theoretical approach to gas entrapment and evolution of aluminium alloys presented in previous papers [1-3]. Thus, a question to be answered was whether the results of gas evolution modelling and theoretical calculation were in agreement with the actual experimental degassing conditions and properties of the processed products.

2. Experimental procedure

The powder used for investigation consisted of a large amount of rapidly solidified P/M alloy, atomized in air, based on the hypereutectic Al–Si–X system containing 20 wt % Si, with an average particle size of 24 μ m [4]. The chemical composition of this P/M alloy, as obtained using atomic absorption spectrophotometry, is Al–18.8 wt % Si–3.2 wt % Cu– 1.1 wt % Mg–0.25 wt % Fe [4].

The processing routes, A and B, applied in this work are shown schematically in Fig. 1. The powder using Process A was cold precompacted to $\sim 65\%$ theoretical density without a can and then compressed to full density and extruded at 350 °C, in a 2 MN horizontal press through a flat die, with an extrusion ratio of 1:20.

Process B requires a vacuum degassing step to remove water vapour and hydrogen. The first step of this process consisted of precompacting the material into a can to about 65% of the theoretical density leaving a proper level of interconnected porosity to allow subsequent degassing to occur efficiently [5]. Each can contained ~ 300 g aluminium powder after compaction. Degassing was carried out covering a wide range of temperatures and times [6].

The second experimental stage of Process B consisted of degassing the Al-20Si-X powder assisted by flushing with a depurative gas such as Ar or N_2 . The temperature and time of the P/M compacts during evacuation and flushing were 400 °C and 1 h, respectively. These parameters were chosen in accordance



Figure 1 Processing routes for aluminium alloy powders used in this work.

with the best degassing conditions obtained by the conventional mode.

The cans with both conventionally and modified degassed compacted powder, still under vacuum, were extruded at 350 °C with an extrusion ratio of 1:20. The extrudates of the non-degassed, conventionally degassed and flushed specimens were subjected to tensile tests at room and elevated temperatures. Prior to tensile testing at elevated temperatures, the products were annealed at the subsequent test temperatures of 100, 200, 300 and 400 $^\circ \rm C$ for 100 h. Tensile tests were performed at a strain rate of 0.5×10^{-2} s⁻¹ using an Instron machine. The extrudates were not heat treated because our previous results [7] indicated that the non-heat-treated Al-20Si-3Cu-1Mg P/M products provide the best strength and elongation at service temperatures above 200 °C. The microstructures were investigated using light microscopy.

3. Results

3.1. Conventional degassing

On the basis of the analysis made over the temperature range in which evolution of H_2 was detected to occur [1, 5] the initial degassing conditions were chosen within a wide range of temperatures and times. This preliminary research was intended to determine the best temperature and time for conventional degassing. A detailed description of the process and results obtained have been reported elsewhere [6].

Based on these results [6] and the research on gas evolution described in [1, 2], the first stage of Process B consisted of extruding the Al–20Si–X compacts degassed at 300, 400 and 500 $^{\circ}$ C for 1 h. Process A, without vacuum degassing, was chosen as a reference level.

The degassing temperatures were chosen with respect to ~ 450 °C which is the temperature where the maximum evolution of hydrogen occurs [1]. The influence of lower and higher degassing temperature than 450 °C on microstructure and tensile properties of the products was investigated.

The results of tensile tests, at room and elevated temperatures, for the extrudates obtained from P/M compacts degassed under the conditions indicated above, compared with that of the non-degassed compacts, are presented in Table I. The code 0 stands for non-degassed material, C301 for canned material degassed at 300 °C for 1 h, C401 for canned material degassed at 400 °C for 1 h and C501 for canned material degassed at 500 °C for 1 h.

These results show a significant increase in tensile properties of the products degassed at 300 and 400 °C with respect to those of the non-degassed compact. However, the properties of the non-degassed sample (0) are comparable at 400 °C and better at 300 °C than those of product C501.

Light microscopy revealed that the degassed materials had coarser microstructures than that of the nondegassed material. The coarsening effect of the preheating temperature, during degassing, was significant above 400 °C. As shown in Fig. 2, the microstructure of the Al-20Si-3Cu-1Mg product degassed at 400 °C for 1 h (C401) properly extruded exhibited the best overall microstructure with low residual porosity and fine silicon crystals. For the degassing conditions (C501) a significant coarsening of silicon crystals lowered the mechanical properties although the porosity disappeared. The microstructure of the non-degassed material (0) was fine, although with visible porosity.

The improvement in the properties of the products C301 and C401 is due to less hydrogen being present in the degassed product, better particle bonding, softer oxides and better oxide redistribution [8]. The product degassed at 400 °C for 1 h (C401) gave the best properties at room and elevated temperatures in comparison with those degassed at lower and higher temperatures, C301 and C501, respectively. A significant improvement in the properties of this product (C401), in the range from room temperature to 400 °C, with respect to those of the non-degassed (0) product was obtained: 16%-40% in ultimate tensile strength, σ_{UTS} , and 67%-457% in elongation, ϵ .

These results show that the properties of the Al-20Si-X P/M products result from a trade-off between the extent of hydrogen evolution during degassing and the coarsening of the microstructure due to a too high degassing temperature. In other words, it is the trade-off between the porosity level and the coarsening of silicon crystals, provided that another hot processing step, such as extrusion, does not cause any additional coarsening of the microstructure.

As far as the extrusion is concerned, a very high temperature, a very high deformation strain rate or a very high extrusion ratio could set the extrusion conditions within the area of severe relative change of microstructure [9].

It can be concluded that the products subjected to a degassing treatment have improved properties

Degassing conditions	Test temperature									
	Room temp.		100 h pre-heating							
			100 °C		200 °C		300 °C		400 °C	
	σ _{uts} (MPa)	в (%)	σ _{uts} (MPa)	з (%)	σ _{UTS} (MPa)	ε (%)	σ _{uts} (MPa)	ε (%)	σ _{uts} (MPa)	ε (%)
0	320	0.7	294	1.0	182	6	101	18	37	32
C301	345	2.5	321	3.7	197	8	117	17	39	37
C401	370	3.9	350	4.1	210	12	120	30	45	60
C501	340	5.0	308	5.6	202	9	93	24	37	51

TABLE I Comparison of tensile properties at room and elevated temperatures of products non-degassed and degassed under different conditions





compared with those of the non-degassed material. The best improvement was obtained for products degassed at 400 °C for 1 h. It is important to point out that the service temperature for Al-20Si-3Cu-1Mg P/M products is in the range 200 to ~ 400 °C so that they can be used up to the threshold service temperature without any risk of further H₂ or H₂O evolution [3] provided that they are processed at optimum degassing conditions.

3.2. Modified degassing

It has been demonstrated that degassing the Al-20Si-3Cu-1Mg powder assisted by flushing with a depurative gas such as Ar or N_2 increases the efficiency of moisture (H₂O) and H₂ evolution, at the prescribed degassing temperature, in comparison with the conventional degassing mode without flushing [3].



Figure 2 Microstructures of extruded products obtained from compacts: (a) non-degassed (0), (b) conventionally degassed at 400 °C for 1 h (C401), (c) conventionally degassed at 500 °C for 1 h (C501). Magnification \times 700.

The tensile values for extrudates obtained from compacts conventionally degassed show that the best properties correspond to the material degassed at 400 °C for 1 h (see Section 3.1). Thus, it was decided to perform tensile testing, at room and elevated temperatures, of products degassed by the modified process (degassing + flushing with Ar or N₂) at 400 °C for 1 h.

Figs 3 and 4 present a comparison of properties, ultimate tensile strength and elongation, at room and elevated temperatures, respectively, for the sample conventionally degassed under optimal conditions (C401) and those samples degassed and flushed with Ar or N_2 at 400 °C for 1 h, CA401 and CN401, respectively.

The products processed with modified degassing gave the following improvement in tensile properties in comparison to those of the conventionally degassed (without flushing) under optimal conditions (Figs 3 and 4): 3%-22%, in ultimate tensile strength, for both Ar- and N₂-flushed samples. In elongation, the improvement was 5%-42% for Ar-flushed samples and 3%-58% for N₂-flushed samples.

These results are in excellent agreement with the modelling of degassing conditions and mode described in previous papers [1–3].

The improvement in properties of the flushed products is due to the fact that modified degassing (degassing + flushing with Ar or N_2) increases the efficiency of moisture (H₂O) and H₂ evolution in



Figure 3 Effect of temperature on the ultimate tensile strength, σ_{UTS} , of extrudates obtained from compacts non-degassed and degassed by different modes. (•) Non-degassed, (\bigcirc) conventionally degassed, (\square) Ar flushed, (\triangle) N₂ flushed.



Figure 4 Effect of temperature on the elongation, ε , of extrudates obtained from compacts non-degassed and degassed by different modes. (\bullet) Non-degassed, (\bigcirc) Conventionally degassed, (\square) Ar flushed, (\triangle) N₂ flushed.

comparison with the products conventionally degassed [3]. The surface bonding energy of N₂ or Ar molecules is generally lower than the H₂O molecules but, during the process of flushing with a depurative gas, the number of collisions of N₂ (or Ar) molecules is $\sim 10^9$ times higher than the number of collisions of the H₂O molecules, for the experimental conditions established in [3]. Thus, any N₂ (or Ar) molecule has more opportunity to occupy any free place in the surface. Once a layer of N₂ (or Ar) molecules is formed on the surface, any other molecule becomes weaker bonded at this layer, which promotes the degassing process [3].

The microstructures of the samples flushed with Ar or with N_2 are, in general, similar to that of the sample conventionally degassed under optimal conditions (C401), Fig. 5. A similar extent of silicon coarsening was observed. Some differences concerned only the level of residual porosity. In general, the residual porosity level was lower for materials degassed and



Figure 5 Microstructures of extruded products obtained from compacts: (a) degassed and flushed with argon at 400 °C for 1 h (CA401), (b) degassed and flushed with nitrogen at 400 °C for 1 h (CN401). Magnification \times 700.

flushed with Ar or with N_2 than that of the sample conventionally degassed under optimal conditions.

4. Conclusions

1. The processing of the Al-20Si-3Cu-1Mg P/M powder must include a degassing step which significantly improves the tensile properties of the extrudates obtained from degassed compacts in comparison with those of the non-degassed ones.

2. The properties of the Al-20Si-3Cu-1Mg P/M products result from a trade-off between the extent of hydrogen evolution during degassing, the porosity level and the coarsening of microstructure.

3. Degassing of the Al-20Si-3Cu-1Mg P/M powder with a modified treatment (degassing + flushing with a depurative gas such as Ar or N_2) gives products with superior tensile properties in comparison to those of the products conventionally degassed (without flushing).

4. At the same strength level, the products of compacts flushed with N_2 (nitrogen) provide better elongation values than those corresponding to products of compacts flushed with Ar.

5. The results of this work are in a very good agreement with the results of the fundamental research on gas entrapment and evolution presented in previous papers. Therefore, it can be concluded that an industrial degassing process can be modelled by a thermodynamic and kinetic approach.

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References

- J. L. ESTRADA, J. DUSZCZYK and B. M. KOREVAAR, J. Mater. Sci. 26 (1991) 1431.
- 2. Idem., ibid. 26 (1991) 1631.
- 3. J. L. ESTRADA and J. DUSZCZYK, *ibid.* in press.
- 4. Idem., ibid. in press.

- 5. J. L. ESTRADA, J. DUSZCZYK, B. M. KOREVAAR and R. YOSHIMURA, in "Science and Technology of Sintering", edited by S. Sōmiya, M. Shimada, M. Yoshimura and R. Watanabe (Elsevier, London, 1988) p. 581.
- J. DUSZCZYK, J. L. ESTRADA, B. M. KOREVAAR, Z. FANG, T. L. J. de HAAN and P. COLIJN, Technical Report for Showa Denko K. K., October 1987 (Japan), Delft University of Technology, The Netherlands.
- 7. J. ZHOU and J. DUSZCZYK, J. Mater. Sci. 26 (1991).
- Y.-W. KIM, in "Progress in Powder Metallurgy", Vol. 43 (1987), edited by C. F. Freeby and H. Hjort (MPIF, Princeton, NJ, 1987) pp. 13-31.
- J. DUSZCZYK, in "Proceedings of the 8th International Light Metals Congress", edited by F. Jeglitsch (Aluminium-Verlag, Düsseldorf, 1987) p. 760.

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