REVISTA DE METALURGIA, 43 (4)
JULIO-AGOSTO, 310-317, 2007
ISSN: 0034-8570

Microstructural characterization and process selection by attributive analysis of eutectic and quasi-eutectic Al-Si alloys for pressure die casting(·)

B. Suárez-Peña*, J. Asensio-Lozano**, J. I. Verdeja-González**,and J. A. Pero-Sanz Elorz***

Abstract In the present study, four alloys from diverse suppliers suitable for obtaining 413.0 metallurgical quality products together with alloy number 2 with an slightly hypoeutectic composition in Silicon were processed. The study focused on the microstructural characterization of the as-received ingots, as well as that of samples obtained from the production process: die cast samples and cold chamber pressure die cast samples. Finally a materials selection process base on microstructural grounds has been put forward as to determine the most suitable starting alloy for industrial production.

Keywords Eutectic aluminium – silicon alloys. Quantitative metallography. Pressure die cast.

Caracterización microstructural y proceso de selección mediante análisis atributivo de aleaciones Al-Si eutécticas y cuasi-eutécticas para fundición a presión

Resumen En el presente trabajo se han analizado cinco aleaciones procedentes de diferentes proveedores aptas para la obtención de componentes de la calidad metalúrgica 413.0, con excepción de la aleación 2 de composición levemente hipoeutéctica. El estudio se centra en la caracterización microestructural de los lingotes en estado de recepción, así como de las muestras obtenidas a partir del proceso de fabricación: muestras coladas en molde metálico y muestras fabricadas mediante fundición a presión en cámara fría. Finalmente se propone un proceso de selección de material basado en consideraciones microestructurales que permite determinar la aleación de partida más apta para la producción industrial mediante fundición a presión.

Palabras clave Aleaciones Al-Si eutécticas. Metalografía cuantitativa. Fundición a presión. Selección de materiales.

1. INTRODUCTION

In pressure die casting technique the molten metal is injected under pressure into a steel die, where it solidifies very rapidly, thus obtaining a component whose geometry and finish are in their final form. Two pressure die casting processes exist: hot chamber and cold chamber. The cold chamber process is used with aluminium alloys: a shot chamber is filled in each cycle, and the chamber and piston are not in continual contact with the molten aluminium.

The conditions of solidification in the pressure die casting process require the use of alloys with excellent castability and resistance to cracking at elevated temperatures, hypoeutectic and quasi-eutectic Al-Si alloys being the most widely used. Among these, the most prominent is the eutectic alloy (413.0) [1].

This type of alloy is characterized by a congruent melting point or a minimum solidification interval, which entails less propensity to microshrinkage[2].

It also presents an attractive combination of cost, strength, hardness and corrosion resistance, together with excellent fluidity and resistance to hot cracking, with a compositional range that is shown in table I.

In order to increase the elevated temperature strength and facilitate ejection as well as minimize

(·) Trabajo recibido el día 19 de septiembre de 2006 y aceptado en su forma final el día 2 de febrero de 2007.
* Department of Materials Science and Metallurgical Engineering, Technical School of Industrial Engineering, The University of Oviedo, Viesques University Campus, Carretera de Castiello de Bernueces, s/n, E-33203 Asturias, SPAIN.
** Department of Materials Science and Metallurgical Engineering, The School of Mines, The University of Oviedo, 13 Independence St., Oviedo, E-33004 Asturias, SPAIN. Corresponding Author: Phone: +34 985 104 302; Fax: +34 985 104 242; e-mail: jasensio@etsimo.uniovi.es
*** Department of Engineering Materials, The School of Mines, The Polytechnical University of Madrid, 13 Rios Rosas St., Madrid, E-28003 Madrid, SPAIN.

Table I. Standardized composition of the 413.0 alloy for pressure die casting expressed in weight percentage[1]

Tabla I. Composición estándar de la aleación 413,0 para colada a presión expresada en porcentaje en peso[1].

Elements		Si	Fe	Cu	Mn	Mg	Ni	Zn	Sn	Bal. (total)
413.0	min	11.0	—	—	—	—	—	—	—	—
	max	13.0	2.00	1.00	0.35	0.10	0.50	0.50	0.15	0.25

soldering to the die faces, alloys with a Fe content between 0.8 wt. % and 1.1 wt. % are usually employed[3 and 4]. Depending on the temperature and the chemical composition of the melt, among other factors, the Fe may precipitate as an intermetallic compound with different morphologies. In alloys close to the eutectic, with Fe concentrations of between 0.5 wt. % and 1.2 wt. %, needle-shaped primary Al_5FeSi crystals (β-AlFeSi) may appear[5], which deteriorate ductility[6]. Furthermore, these iron-rich needles could form early on during solidification and are usually associated with the shrinkage voids present in the solidified material[3].

The objective of the present work is to formulate a materials selection process by means of the application of an attributive analysis based on microstructructural analysis for components made of eutectic and quasi-eutectic Al-Si alloys, processed by high pressure die casting. The work has been developed at the existing facilities that the Thyssen Krupp Güss has at the factory located in Asturias (Spain). The present study reports on the microstructure evolution of the five alloys used at different stages of the making process, i.e.: as-received ingots, after industrial degassing and atmospheric solidification in preheated dies and finally, after being processed under high pressure die casting.

2. EXPERIMENTAL PROCEDURE

2.1. Materials

To carry out the study, four alloys from diverse suppliers suitable for obtaining 413.0 metallurgical quality products together with alloy number 2 with an slightly hypoeutectic composition in Silicon were processed. The nominal compositions of these are shown in table II. These melts were processed by means of the existing pressure die casting technology employed at the Thyssen Krupp Guss factory (Spain)[7 and 8].

2.2. Pressure die casting process

The industrial procedure followed in all the tests carried out on the ingots from the five suppliers was:

— Samples were taken of the as-received ingots for metallographic characterization.
— Amounts of around 700 Kg were then poured from the melting furnace into the treatment ladle and subsequently degassed with nitrogen.
— Samples were then poured into a metallic mould preheated to 340 °C and left to solidify for subsequent metallographic observation.
— The melts were then transferred to a maintenance furnace from the treatment ladle, where it was kept at temperature of 675 °C. A predetermined amount of liquid melt was subsequently introduced into the injection chamber where it was solidified under high pressure. Finally a sample was cut from the component in order to carry out the metallographic characterization of the finished product.

2.3. Metallographic characterization

Metal samples for metallography were prepared by conventional mechanical methods and etched with

Table II. Compositions of the as-received 413.0 alloy ingots

Tabla II. Composiciones de los lingotes de la aleación 413,0 en estado de recepción

Alloy	Si	Fe	Cu	Mn	Mg	Ni	Zn	Pb	Ti	P	Cr
1	12.32	0.72	0.03	0.14	0.030	0.030	0.02	0.010	0.006	—	0.008
2	10.10	0.93	0.02	0.10	0.015	0.009	0.01	0.002	0.064	—	0.010
3	11.59	0.82	0.04	0.24	0.009	0.001	0.02	0.008	0.012	—	0.004
4	12.26	0.75	0.04	0.18	0.020	0.006	0.02	0.010	0.100	0.001	0.004
5	12.10	0.80	0.07	0.18	0.043	0.009	0.06	0.020	0.021	—	0.013

0.5 % HF aqueous thereafter. The equipment used was a NIKON EPHIPHOT metallographic bench for optical microscopy (LOM) and a JEOL JSM 6100 scanning electron microscope (SEM) connected to an Oxford Inca Energy 200 dispersive energy microanalyzer.

The determination of the volume fraction and dendrite size of the primary silicon particles, on the one hand, and of the volume fraction of α-Al phase, on the other, were conducted following the quantitative metallogrphic techniques: point counting and mean linear intercept analysis[9-12]. At least 165 characteristics were measured to evaluate the volume fractions and some 350 characteristics for the particle size. The relative error of the determination for each parameter did not exceed 8 %.

3. RESULTS AND DISCUSSION

3.1. As-received ingots

Figure 1 shows the micrographs of the as-received ingot alloys from the five suppliers. The microstructure observed in all the ingots consisted of primary aluminum dendrites, as well as silicon and the eutectic phase. The existence of these phases contravenes the equilibrium diagram of the Al-Si system and reflects both the complexity of the solidification process under industrial conditions as well as the influence of the different degrees of purity of the melts being employed. Gruzsleski[13] proposes a possible explanation of the solidification process based on nucleation and segregation, as depicted in figure 2: silicon crystals are the first to nucleate below the stable eutectic temperature, at some degree of undercooling (point A). As the cuboids grow, the surrounding liquid becomes depleted in silicon. This implies the formation of Al halo around the former particles, which takes places with further decrease in temperature. As a result point B in the phase diagram is arrived at, and formation of primary dendrites occurs. The small solubility of Si in Al dictates a new displacement of the composition of the liquid to point C, during the solidification of α-Al. On doing so, the liquid fall into a region denominated "coupled zone", where the eutectic reaction develops into a lamellar structure of alternating Si and α-Al platelets.

The results obtained after applying quantitative metallographic techniques (Table III) indicate that, although the microstructure features evaluated in all the ingots is similar, alloy 4 is the most favorable alloy in view of a selection process that simultaneously rewards a low volume fraction and a small size of Si cuboids, which act as stress raisers propitiating a downgrading of the mechanical properties of the components. Gruzsleski[14] has shown that the refining of silicon is generally achieved by means of the phosphorous. P reacts with aluminium in the liquid state and produces a fine dispersion of aluminium phosphide (AlP), the crystalline structure of which is very similar to that of silicon. It therefore serves as an effective heterogeneous nucleation site for this phase.

Comparison of tables I and II reveals that the Fe contents fall within the limits given by the standard for 413.0 in all the analyzed compositions. This element reduces the tendency to microshrinkage and favors ejection of the component from the die. However, it is prone to precipitate in the form of crystals with an acicular morphology (α-AlFeSi), highly unfavorable from the point of view of

Figure 1. Light optical micrographs of the 413.0 as-received ingot: (a) Alloy 1; (b) Alloy 2; (c) Alloy 3; (d) Alloy 4 and (e) Alloy 5.

Figura 1. Micrografías ópticas de los lingotes de calidad 413.0 en estado de recepción: (a) Aleación 1; (b) Aleación 2; (c) Aleación 3; (d) Aleación 4; (e) Aleación 5.

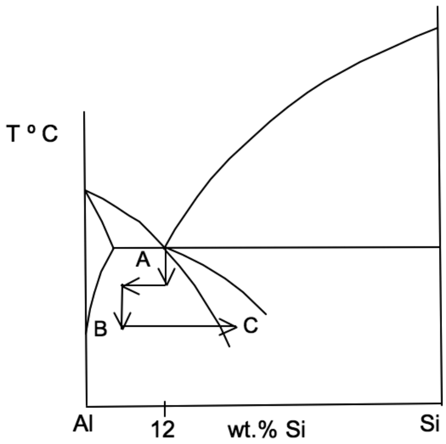

Figure 2. Solidification route for 413.0 aluminium alloys[13].

Figura 2. Secuencia de solidificación de la aleación 413.0 [13].

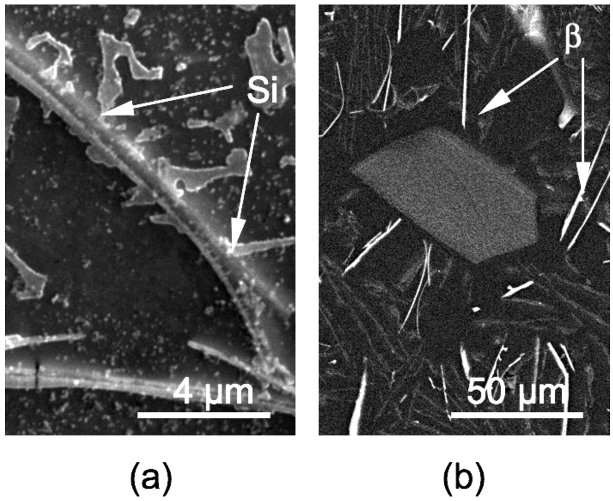

Figure 3. SEM micrographs of intermetallic compounds in Alloy 5: (a) α-AlFeSi needle serving as a substrate for heterogeneous nucleation of eutectic silicon (secondary electron image); (b) isolated, needles homogeneously distributed in the eutectic matrix (back-scattered image).

Figura 3. Micrografías electrónicas de barrido de los compuestos intermetálicos en la aleación 5: (a) Aguja de α-Al-FeSi actuando como agente de la nucleación heterogénea para el silicio eutéctico (electrones secundarios); (b) agujas del intermetálico , aisladas, distribuidas homogéneamente en la matriz eutéctica (imagen de electrones retrodispersados).

mechanical properties. Fig. 1 shows the existence in all cases of these platelets, seen as needle-shaped intermetallic compound in the plane of polish. SEM micrographs, show that the β-phase needles are likely nucleating agents of the eutectic phase. In effect, figure 3a), illustrates of this mechanism, where the eutectic silicon have developed onto the sides of the ,- platelets. On other occasions, the β-phase needles appear isolated, thicker and located in the matrix, intermingled in the Al-Si eutectic, though presenting a smaller size (Fig. 3b)) [5]. Manganese transforms the needle-shaped compound into a dendritic intermetallic compound (α-AlFeSi) by substitution of the Fe atoms in its crystalline structure. From the viewpoint of the mechanical properties of the alloy, this morphology, known as "Chinese script", is not so detrimental. The most adequate Fe/Mn ratio to stabilize the α-AlFeSi phase is 2/1 [15]. However, this ratio was not found in any of the analyzed alloys.

Titanium is an element used to refine the dendrite size of the α-Al. Observation of figure 1 shows that one of the finest dendrite sizes of the α-Al corresponds to the alloy with a higher titanium content: alloy 4.

Table III. Volume fraction (V_v(cub)), and particle size (d(cub)) of cuboidal silicon in all stages of the processing route analyzed. And volume fraction of α-Al dendrites $V_v(\alpha)$, after being processed by high pressure die casting.

Tabla III. Fracción en volumen (V_v(cub)) y tamaño de partícula (d(cub)) de silicio cuboidal en todos los estados del proceso analizados. Y fracción en volumen de dendritos de Al-α ($V_v(\alpha)$) tras procesado mediante colada a presión.

Alloy	As received ingot		Chill die casting		Pressure die casting				
	V_V (cub) %	d (cub) µm	V_V (cub) %	d (cub) µm	V_V (cub) %	d (cub) µm	$V_V(\alpha)$ %	Q_j	$\frac{Q_{max}}{Q_j}$
1	1.02	16.1	2.02	24.0	0.71	3.8	41.38	92.3	1.02
2	0.58	26.4	1.39	27.7	1.11	5.1	44.33	76.8	1.23
3	1.95	22.1	2.36	23.1	1.84	4.5	37.16	72.0	1.31
4	0.86	14.5	1.43	59.0	0.70	3.8	43.95	94.4	1.00
5	1.27	25.9	0.51	36.9	1.18	3.5	46.23	93.2	1.01

3.2. Chill cast samples

After degassing with N_2 and solidification under atmospheric conditions in preheated metallic mould, the resulting microstructure presents differences with respect to the as-received ingots (Fig. 4): a thickening of all the phases present is observed in all cases. Quantitative metallography corroborated these results, an increase being observed not only in the size of the cuboids, but also in their volume fraction. The most pronounced increases as regards the diameter of the cuboids corresponds to alloys 4 and 5, while the volume fractions increased in all cases, with the exception of alloy 5, which suffered a 60% reduction.

3.3. Pressure die casting components

After subjecting the alloys to pressure die casting, the resulting microstructure is made up of fine dendrites of the α-Al with a quasi-equiaxial morphology, together with very scarce, extremely fine cuboids of silicon that are only visible at a magnification of 1000x, as well as a eutectic phase with silicon in the form of fine needles (Fig. 5). This is due to the faster cooling rate , which not only refines the Si particles, but also reduces the interdendritic space (DAS) of α-al favoring its cell-like growth thus reducing the level of porosity and of microshrinkage[14]. Normally in the Al-Si alloy, the crack initiates at the interface of the hard silicon

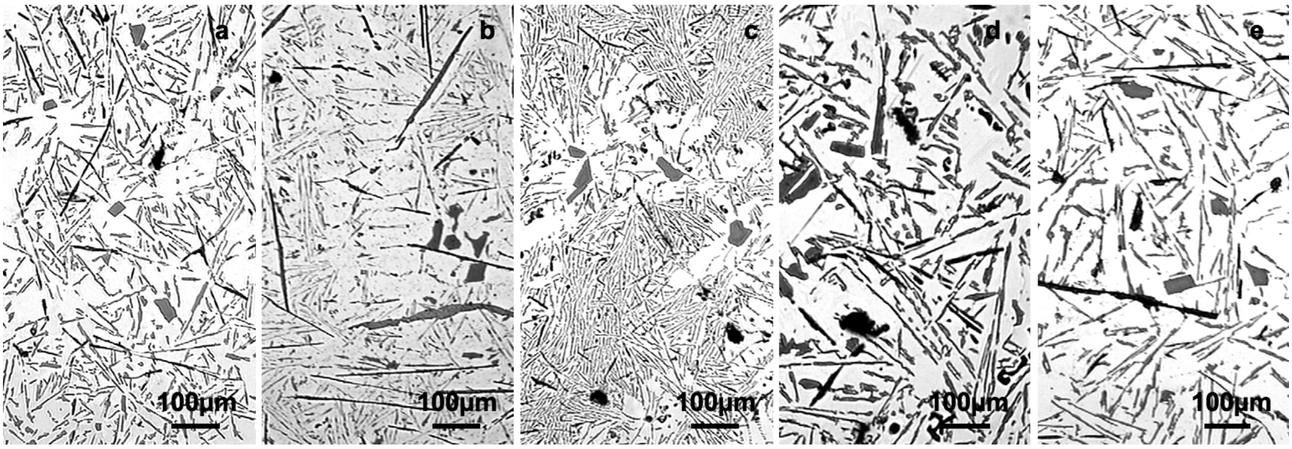

Figure 4. Light optical micrographs of the 413.0 die preheated at 340 ºC cast alloys: (a) Alloy 1; (b) Alloy 2; (c) Alloy 3; (d) Alloy 4 and (e) Alloy 5.

Figura 4. Micrografías ópticas de aleación 413.0, procedentes de enfriamiento en molde metálico precalentado a 340 ºC: (a) Aleación 1 ;(b) Aleación 2; (c) Aleación 3; (d) Aleación 4; (e) Aleación 5.

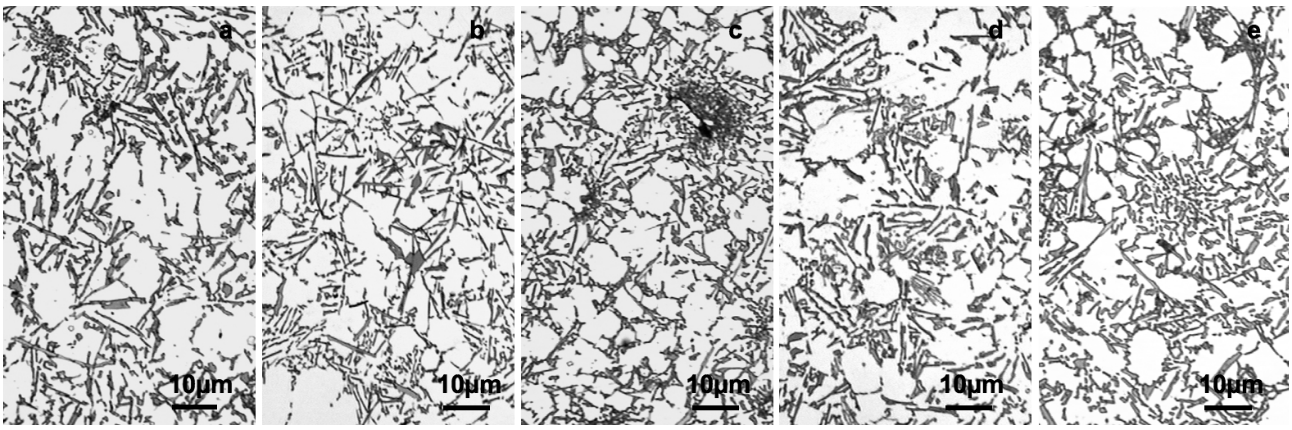

Figure 5. Light optical micrographs of the different alloys after being processed by the pressure die casting method: (a) Alloy 1; (b) Alloy 2; (c) Alloy 3; (d) Alloy 4 and (e) Alloy 5.

Figura 5. Micrografías ópticas de las aleaciones tras procesado mediante la técnica de fundición a presión: (a) Aleación 1 ;(b) Aleación 2; (c) Aleación 3; (d) Aleación 4; (e) Aleación 5.

particles and the soft matrix and then propagates through the matrix (Figs. 6 a), b) and c)). If porosity and/or solidification shrinkage is present in the interdendritic region, the cracks find their path along these voids[8 and 16]. This has been evidenced from the SEM micrograph in figure 6 d), which shows the easy crack path through the voids. Vorren et al found that the β phase solidifies both in grain boundaries as well as in the interdendritic regions, therefore aiding the nucleation of cracks and their rapid propagation by acting as stress raisers[17]. In this work no evidences of the development of beta phase needles were found acting as stress raisers in the fracture facets analyzed.

The quantitative metallographic determinations showed that, after subjecting the alloys to pressure die casting, the size of the Si cuboids decreased appreciably in all cases. The finest cuboids may be expected to correspond to the alloy with the higher content in P (alloy 4). However, the size of these cuboids was found to be very similar to that of the other alloys that did not contain P (alloys 1 and 5). This leads to the consideration that low contents in P in the starting ingot are innocuous in the refining of Si cuboids, after the industrial treatment by means of the pressure die casting technique[18]. As regards the volume fraction of α-Al, the interest of this parameter lies in the fact that it is capable of providing plastic confinement zones ahead of the cracks. In this respect, alloys 5, 2 and 4 would be the most favorable, in this order, to judge by the results of table III.

3.4. Attributive analysis of studied alloys apt for pressure die casting

Finally, in view the above, to evaluate which alloy is the most suitable for the industrial processing of components by high pressure die casting with improved fatigue resistance, an attributive analysis was used. This methodology of analysis consists in minimizing the size and volume fraction of the silicon cuboids and maximizing the volume fraction of the α-Al, assigning these a relative importance of 50 % to the initiation of cracks which mainly takes place at coarse Si cuboids and therefore depends on the particle size. The remaining percentage is distributed as follows: a 33.3 % corresponds to tip blunting at the tougher α-Al phase, and a remaining 16.6 % attributed to the stressed volume located around cuboidal Silicon [8 and 19-22]:

$$Qj = 100 \times \left[\frac{1}{6}\left(\frac{(Vv(cub))_{min}}{(Vv(cub))_j}\right)+\frac{1}{2}\left(\frac{(d(cub))_{min}}{(d(cub))_j}\right)+\frac{1}{3}\left(\frac{(Vv(\alpha))_j}{(Vv(\alpha))_{max}}\right)\right] \quad (1)$$

The formulation (1) responds to process for materials selection being put forward by M.F. Ashby and J.A. Pero-Sanz[19 and 20]:

$$Q_j = 100 \times \left(\overbrace{w_1}^{\text{weight coefficient}} \cdot \underbrace{\frac{K_j}{K_{max}}}_{\text{favourable parameter to maximize}} + \ldots + \overbrace{w_n}^{\text{weight coefficient}} \cdot \underbrace{\frac{M_{min}}{M_j}}_{\text{unfavourable parameter to minimize}} \right) \quad (2)$$

$$\sum_{i=1}^{n} w_i = 1$$

When a material is to be chosen with multiple constrains for a given application, the above formulae

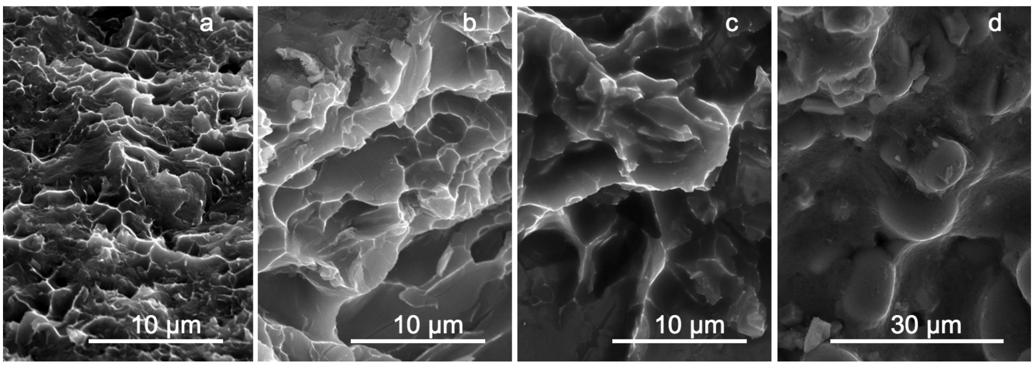

Figure 6. SEM micrographs of the fracture surfaces in bending fatigue tests: (a) and (b) for alloy 1, and (c) for alloy 4, after processing by pressure die casting, in samples showing decohesion facets at former Si particles; and (d), in alloy 4, after processing by atmospheric die casting, on a sample showing a fracture facet at a microshrinkage.

Figura 6. Micrografías MEB de las superficies de fractura tras ensayos de fatiga por flexión de: (a) y (b) en la aleación 1, y (c) en la aleación 4, mostrando facetas de descohesión en antiguas partículas de Si en muestras obtenidas por colada a presión; y (d) en la aleación 4, ilustrando la superficie de fractura en un microrrechupe de una muestra enfriada en molde metálico.

(2) shows how to simply compose an index which ponders favourable (K_j) and unfavourable (M_j) parameters, according to given weight coefficients (w_i). After each value of Q_j is derived for every candidate material, it is possible to rank the alloys according to the value so obtained. As a result, among the candidate materials it is possible to arrange the different Q_j values so as to derive the highest one, ie.: Q_{max}. By dividing Q_{max}/Q_j it is possible to categorize alloy *i* with respect to the best in the list. The weight coefficients w_i have been reported in a Private Communication of Thyssen Krupp Company in their work conducted at the factory located in Mieres (Asturias) and are the result of the analysis for improved fatigue resistance[21].

Table III shows the results of the merit index (Q_j), as well as the ratio Q_{max}/Q_j. In view of the results, the following order of suitability may be established in the following order: Alloy 4, then Alloy 5, then Alloy 1, then Alloy 2 and the least suitable being Alloy 3.

The methodology so described for the attributive analysis based on microstructural grounds, as well as the rational criteria behind the specific values being chosen for the weight coefficients, are validated by the low cycle – high stress bending fatigue tests conducted on actual components (escalator steps) made of the alloys under analysis. The description of the test procedure is described elsewhere[7 and 8]. The results in table IV corroborate the order of goodness found in table III.

4. CONCLUSIONS

A method for quantifying the most suitable Al-12Si alloy for pressure die casting among different commercial alloy candidates has been put forward. The ranking methodology of the alloys consists in the application of an attributive analysis where some of the quantitatively determined microstructural features are maximized (volume fraction of α-Al) and others (volume fraction and particle size of Si cuboids) minimized. The method includes the application of weight coefficients based on practical experiences derived from the observed correlations of the above mentioned microstructure parameters with the fatigue resistance tests under bending conditions.

Acknowledgements

The authors wish to express their special thanks to the University of Oviedo Vice-rectorate for Research for economic support in the preparation of the draft version of this paper.

Table IV. Results of low cycle-high stress bending fatigue for escalador steps processed by high pressure die casting from the studied alloys. Where: $U_{T,b}$ represents the total energy involved in the fracture of the component; F_R, is the rupture force; and δ_R, is the maximum deflection at breakage.

Tabla IV. Resultados obtenidos tras ensayar a fatiga de "bajo número de ciclos" por flexión, peldaños de elevación fabricados mediante fundición a presión a partir de las aleaciones objeto de análisis. donde: $U_{T,b}$ es la energía consumida en la rotura del material; F_R, la carga de rotura, y δ_R, la flecha máxima experimentada por el material a rotura.

Aleación	$U_{T,b}$ (J)	F_R (KN)	δ_R (mm)
1	202,81	23,41	13
2	140,36	19,96	6
3	121,18	19,50	10
4	254,90	29,55	24
5	225,11	29,40	22

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