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Statistical analysis of the tensile strength of an Al_2O_3 short-fibre-reinforced aluminium composite

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Metal matrix composites are presently being introduced in various industrial applications; specifically δ-alumina short-fibre-reinforced aluminium composites are already employed by some Japanese companies for the production of car pistons. Many studies on the fundamental properties of these composites can be found in the literature. However, in spite of what would be expected from an industrially employed material, some relevant aspects still remain to be studied. One of these important aspects scarcely found in the literature is the reliability of the material. This paper presents a statistical evaluation of the tensile strength of a δ-alumina short-fibre-reinforced aluminium alloy by means of Weibull statistics, in order to determine the reliability and repeatability of the material.

1. Introduction

Light metal matrix composites reinforced with ceramic particles and short fibres are increasingly being introduced in structural applications because they can offer advantageous properties, such as wear resistance and high-temperature capabilities, at competitive prices [1]. The automotive industry is a pioneer customer of them. Specifically, some Japanese companies have already introduced aluminium alloy pistons partially reinforced with δ-alumina short fibres in the ring land area, improving their wear resistance and the mechanical durability at elevated temperatures [2]. The working temperature in the bowl edge of the engine pistons may approach 500 °C, and aluminium alloys suffer a rapid loss of strength at temperatures over 150 °C, while the strength of the composites remain at levels near to those of room temperature [3]. In addition to the high temperatures, the crown is subjected to severe thermal and mechanical cyclic stresses which give rise to cracking. Alumina short-fibre-reinforced composites seem to extend lifetime under these severe conditions [4].

Although many papers in the literature focus on the mechanical behaviour of these types of composites, few of them mention the reliability of the material and the repeatability of fabrication. The aim of the present work was to study the reliability of an alumina short-fibre-reinforced composite specifically developed for the production of car pistons. The material was supplied by the company FOARSA (Foarsa presently Sidenor, Paseo de Alejandro Calonge 1, Reinosa 39200, Spain). It consists of aluminium alloy AS12 UNG reinforced with 15% Saffil^{TM} (ICI, Runcorn, UK) alumina short fibre. Owing to the simplicity and typical use in the evaluation of reliability of materials, Weibull statistics was selected for the present study. The properties evaluated are the room-temperature ultimate tensile strength and the yield strength of both the composite and the unreinforced alloy. A microstructural and fractographic analysis has also been performed in order to determine the critical features of the composite, and to compare them with those of the alloy.

2. Experimental procedure

The materials consisted of 160 mm diameter circular discs produced by squeeze casting and composed of two zones: the upper part is made of aluminium–silicon eutectic alloy AS12 UNG (composition shown in Table I) 15 mm thick, and the lower part is of 15% alumina short-fibre ("Saffil" preforms supplied by Vernaware Ltd, Bolton, UK)-reinforced aluminium alloy AS12 UNG, 20 mm thick. The discs were produced and supplied by Foarsa. Microstructural observation and analysis of the phases present in the alloy and the composite was done by optical and scanning electron microscopy in a Jeol microprobe, aided by X-ray energy dispersive analysis.

Four discs were cut and machined into cylindrical tensile specimens, both from the composite and the

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TABLE I AS12 UNG alloy composition

Alloying elements (%)											
Si	Cu	Mg	Ni	Ca	Fe	Mn	Pb	Sn	Ti	Zn	Al
12.05	1.24	0.98	1.05	0.002	0.36	0.04	0.002	0.001	0.008	0.009	Bal.

unreinforced alloy, for the determination of the ultimate tensile strength (UTS) and the yield strength (YS). The specimens and the tests were carried out according to ASTM D 3552-77 at a crosshead speed of 5 mm min^{-1}. The deformation was measured by axial extensometry over a 25 mm gauge length.

In order to check the homogeneity of the material in each disc and the reproducibility from disc to disc, two of the discs were cut following the schedule of Fig. 1a, and the other two follow the schedule of Fig. 1b. In all 48 tensile specimens of each material were tested.

The tests were carried out in randomized order, and the results were studied by Weibull analysis [5], according to the following two-parameter equation

$$\ln\{\ln[1/(1-P)]\} = K + \ln \sigma^m \qquad (1)$$

where K is a constant, σ is the evaluated property (the ultimate and the yield strength in this case), P is the probability, and m is the Weibull modulus. Plotting the left part of the equation versus $\ln \sigma$ should give a straight line, its slope being the Weibull modulus. The higher the modulus, the narrower is the dispersion of the results, and the more reliable the material.

A study of the composite results in randomized groups was also made in order to determine the error committed when a small number of specimens is tested. Therefore, 7 groups of 4 data, 4 groups of 10 data, and 3 groups of 20 data, all randomly selected, were formed from the 48 tested specimens, and the average values calculated. Finally, in order to determine the repeatability of the materials characteristics from batch to batch, the results were evaluated in groups corresponding to each disc.

Determination of the most relevant and critical features in the composite and the unreinforced alloy was performed by fractographic observation in a Jeol microprobe.

3. Results

3.1. Microstructure

The microstructure of the alloy is composed of multiple phases, as corresponds to the highly alloyed composition. Fig. 2 shows an unetched micrograph, where many different phases can be clearly distinguished. Most of them are complex compounds of different proportions of aluminium, silicon, nickel, copper and iron. The lightest phases contain higher proportions of copper and nickel, while the darkest ones are enriched in silicon. The black phase visible in the micrograph of Fig. 2 corresponds to the compound Mg_2Si.

The microstructure of the composite is shown in Fig. 3. Although the nature of the phases is the same as in the alloy, some relevant differences are observed: the size of the phases is much smaller, the Mg_2Si phase is now precipitated around the fibres, and also some primary silicon precipitates are sporadically detected (grey regular particles visible in Fig. 3), while almost none could be seen in the alloy.

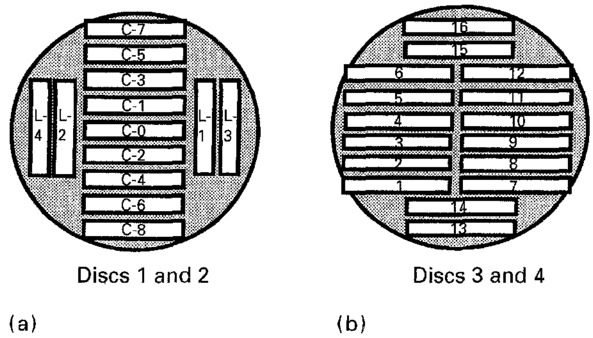

Figure 1 Schedule of the specimens from each disc.

Figure 2 Microstructure of the alloy (×400).

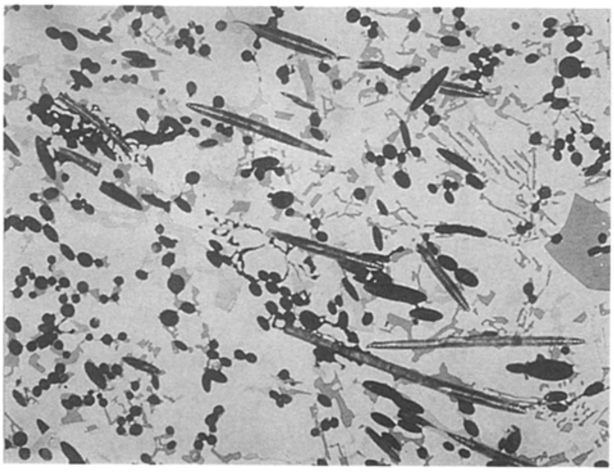

Figure 3 Microstructure of the composite (×400).

Observation of transverse sections of the several discs shows an apparently homogenous distribution of phases and fibres in both the composite and the unreinforced alloy. Nevertheless, in the unreinforced alloy the size of the phases is smaller in the upper part (where refrigeration was located) than in the lower part.

Two isolated huge inhomogeneities were detected in a couple of sections. One was a large zone with no fibres at all due to a deficient fibre distribution, shown in Fig. 4. The other one is a zone with an extremely large number of primary silicon precipitates (Fig. 5). Although these features cannot be considered representative of the composite structure, they are huge defects that can be present in many discs due to their large size.

3.2. Mechanical tests results

The mean UTS and YS of each disc and of the entire composite population are shown in Fig. 6, as well as the coefficient of variance (= standard deviation divided by the mean and multiplied by 100). The results of the aluminium specimens are shown in Fig. 7. Both the UTS and YS of the composite and the unreinforced alloy specimens did not show any dependency on the zone of the disc from which they were extracted.

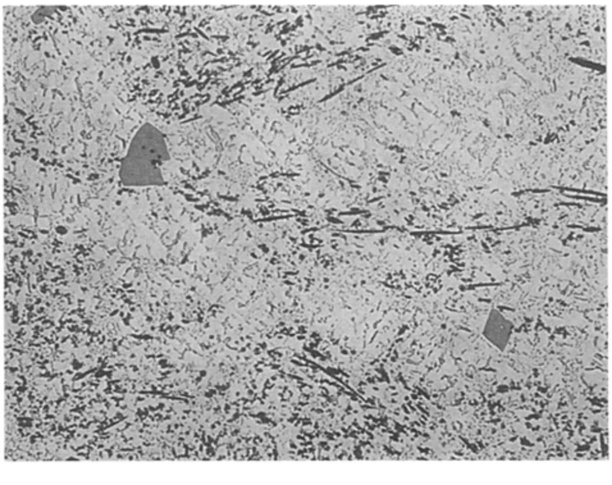

Figure 4 Inhomogeneous fibre distribution (× 100).

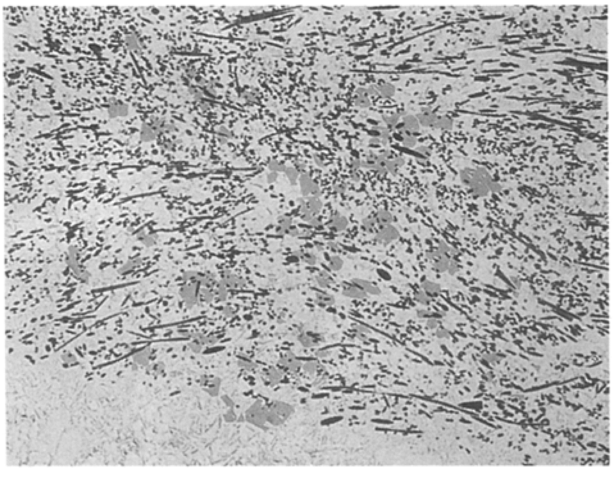

Figure 5 Clusters of silicon precipitates (× 100).

3.3. Statistical analysis

The Weibull plots of the UTS and YS of the composite and the unreinforced alloy populations are shown in Fig. 8, and the Weibull modulus of both properties can be seen in Table II.

In order to determine the reliability of the results when only a small number of specimens are tested, groups of random data were formed. The average UTS and YS values of these groups, and of the groups formed with the data from each disc, are shown in Fig. 9. The percentage difference between each group average and the average of the total population ("error") are marked with vertical bars on each group value.

3.4. Fractographic analysis

Several fractured specimens were observed in order to find the most critical features or defects in the composite. The presence of clusters or bundles of fibres perpendicular to the load direction appeared to be the fracture origin in many of the "weak" specimens, especially when they are located close to the surface of the specimen. As an example, Fig. 10 exhibits the fracture of the specimen Disc2-C4, where the presence of a bundle of fibres perpendicular to the load can clearly be observed. Other "weak" specimens from different discs and locations presented similar defects in their fractures. The stronger specimens did not show special features that could be assigned as fracture origins,

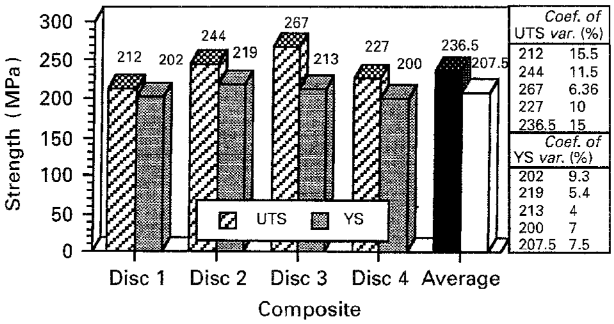

Figure 6 Mean values and coefficient of variance of composite populations.

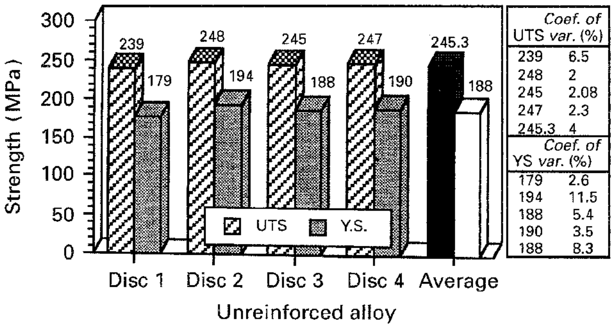

Figure 7 Mean values and coefficient of variance of alloy populations.

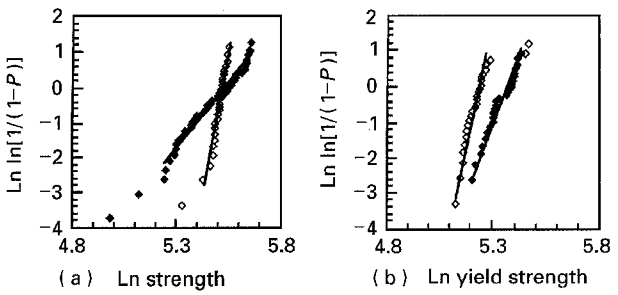

Figure 8 Weibull plots of (a) the ultimate tensile strength and (b) the yield strength of (◆) the composite and (◇) the unreinforced alloy.

TABLE II Weibull modulus of the composite and the unreinforced alloy

Property	Weibull, modulus, m	
	Composite	AS12 UNG
Ultimate strength	7.46	35
Elastic limit	14	24.5

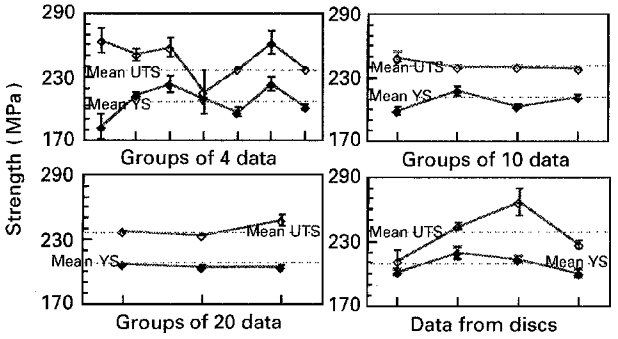

Figure 9 (◇) UTS and (◆) YS of various groups of data, and error percentage with respect to the total population average.

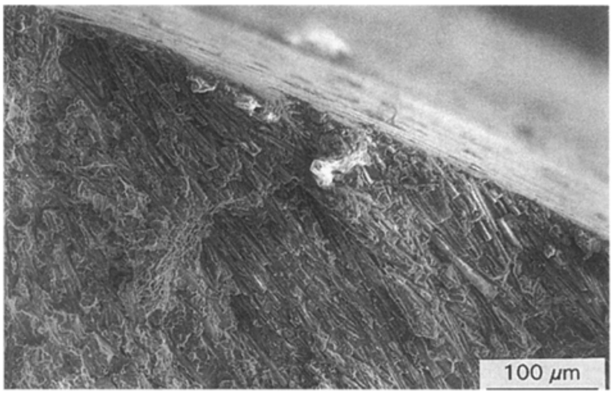

Figure 10 Fracture origin of a composite tensile specimen.

except for a large silicon precipitate detected in specimen Disc 3-6, which broke at 229.36 MPa.

The fractographic observation of the unreinforced specimens showed a macroscopically brittle, microscopically ductile behaviour, as could be expected from the highly alloyed, untreated, aluminium alloy.

4. Discussion

The mean UTS and YS values for both the unreinforced alloy and the composite are in accordance with the data found in the literature. A UTS of 190 MPa and YS of 150 MPa are usually expected from the unreinforced alloy [6], which is largely overcome by the material studied in the present work. In the case of the composite data, various UTS values are found in similar composites (same alloy, alumina–silica short-fibre reinforcement, and volume fractions from 15%–18%) ranging from 185–273 MPa [7–9].

A first comparison between the data from the composite and those from the unreinforced alloy show that, although the room-temperature UTS of the composite is slightly lower (around 10 MPa difference) the YS increases favourably (around 20 MPa increment). This is due to the effect of the fibrous ceramic reinforcement, which confers rigidity and, therefore, increments the YS, while the UTS can even decrease slightly with respect to the unreinforced base alloy. This behaviour changes at higher temperature, when the properties of the alloy rapidly fall, while the composite ones remain close to the room-temperature values [10].

The dispersion of the composite population, however, is significantly larger than that of the alloy, and the variation in the results from different batches is also larger in the composite in spite of having been produced at the same time and, therefore, conditions. The comparison of the results from the centre of the discs (see Fig. 1), C-0/C-1/C-2, from the middle part, C-3/C-4/and 1–12, and from the outer zone, L-1/L-2/L-3/L-4/and 13–16, showed that the strength distribution and the scattering are uniform, and no macroscopic stronger or weaker zones were found in the material.

The strength distributions of Fig. 8 clearly indicate the high reliability of the unreinforced alloy, with Weibull modulus of 35 and 24.5 for the UTS and the YS, respectively, showing a large margin on the mean strength for design of components, while the strength distributions of the composite specimens give Weibull modulus close to those offered by typically brittle materials [11]. This means that those applications that require certain strength levels should be overdesigned, and an important safety coefficient ought to be applied. Special care was taken during the production of the discs in order to guarantee similar production conditions. Therefore, from both the UTS/YS and the Weibull modulus of the unreinforced alloy, it can be inferred that the processing conditions are adequate, and that the dispersion of the composite results is mainly due to other factors.

The intrinsic heterogeneity observed in the microstructural and fractographic analysis is an important factor that is conducive to a large scattering in the results. The large inhomogeneity in the distribution of the fibres (which was initially inadequately considered to be uniform) and the existence of defects such as those of Fig. 5 (zones with almost no fibres) and Fig. 1 (fibres forming clusters or bundles) is finally responsible for the high scatter of the results. Additionally, the bundles of fibres located perpendicular to the applied load appeared to be the fracture origin of many of the weakest specimens, and this indicates the strength increment that could be obtained if they were avoided. The large presence of silicon precipitates in the composite could also help to increase further the scattering. Primary silicon precipitates are almost unavoidable when the composite base alloy is a eutectic Al–Si, because they are produced by the reaction of the SiO_2 on the fibres with the magnesium of the alloy producing Mg_2Si/MgO around the fibres and silicon precipitates [12]. It also seems possible that the SiO_2 could react with the aluminium, producing Al_2O_3 and primary silicon [13].

The results of the groups formed with random data show that the error that can be produced when only four specimens are tested is in the range from 10%–20% with respect to the average of 48 specimens. The groups formed with ten specimens showed errors below 5%, and the groups of 20 specimens gave also errors below 5%. Hence, care must be exercised when evaluating these properties with a small number of specimens.

Automotive pistons are usually partially reinforced with small preforms and, consequently, better homogeneity can be expected than in the present study. Nevertheless, the scattering of the results will still be much larger than in the unreinforced alloy, being the avoidance of large defects, especially fibre bundles perpendicular to the load direction, and the refinement of the preform fibre distribution the most important factor in the enhancement of the materials tensile properties and reliability.

5. Conclusions

1. Composites made of aluminium alloy AS12 UNG–15%Saffil alumina short fibre produced by squeeze casting, exhibit a UTS of 236.5 MPa and a YS of 207.5 MPa. The UTS of the unreinforced alloy produced together with the composites is 245.3 MPa, and its YS 188 MPa.

2. The scattering of the composite results is very large, with a UTS Weibull modulus of 7.4, and a YS Weibull modulus of 14. The corresponding moduli for the unreinforced alloy are 35 and 24.5, respectively.

3. The most important factor for the large scattering of the results is the non-uniform fibre distribution of the preform, and the presence of fibre bundles.

4. The fracture origin in the "weakest" specimens is usually a bundle of fibres perpendicular to the load direction.

5. More reliability in the results could be obtained if the distribution of the fibre in the preform was optimized, and defects such as bundles of fibres were eliminated.

6. A minimum of ten specimens is recommended for the determination of the UTS and YS. A smaller number of specimens could lead to an important degree of error.

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References

1. R. K. EVERETT and R. J. ARSENAULT, "Metal Matrix Composites: Mechanisms and Properties" (Academic Press, San Diego, CA, 1991).
2. T. DONOMOTO, F. FUNATAMI, N. MIURA and N. MIYAKE, SAE paper 832052 (Michigan, March 1983).
3. S. B. LASDAY, *Ind. heating* March (1987) 20.
4. J. DINWOOODIE, E. MOORE, C. A. J. LANGMAN and W. R. SYMES, in "Proceedings of ICCM-V", edited by W. C. Harrigan *et al.* (Metallurgical Society, San Diego, 1985) pp. 671.
5. G. E. DIETER, "Mechanical Metallury", 3rd Edn (McGraw-Hill Series in Materials Science and Engineering, Singapore, 1986) pp. 370.
6. "Nomenclature internationale des alliages dâluminium de fonderie", 4th Edn (Techniques des industries de la fonderie, Paris, 1986).
7. L. ACKERMANN, J. CHARBONIER, G. DESPLANCHES and H. KOSLOWSKI, in "Proceedings of ICCM-V", edited by W. C. Harrigan *et al.* (Metallurgical Society, San Diego, 1985) pp. 687.
8. F. K. CHI, R. D. MAIER, T. W. KRUCEK and P. M. BOYMEL, in "Proceedings of ICCM & ECCM II", Vol. 2, edited by F. L. Mathews *et al.* (Elsevier Applied Science, London, 1987) pp. 2449.
9. J. BÄR, H. G. KLUSSMANN and H.-J. GUDLADT, *Scripta Metall. Mater.* **29** (1993) 787.
10. T. W. CLYNE, M. G. BADER, G. R. CAPPLEMAN and P. A. HUBERT, *J. Mater. Sci.* **20** (1985) 685.
11. J. M. TING, Y. KO and R. Y. LIN, *Am. Ceram. Soc. Bull.* July (1991) 1167.
12. A. MUÑOZ, J. GOÑI, J. F. LICEAGA, in "Proceedings of the 12th International Symposium on Materials Science", Risø National Laboratory Denmark, 1991, edited by N. Hansen *et al.* (Risø, Denmark, 1991) pp. 543.
13. C. FRIEND, R. YOUNG and I. HORSFALL, in "Proceedings of EACM 3", Bordeaux 1989, edited by A. R. Bunsell *et al.* (Elsevier Applied Science, NY, 1989) pp. 227.

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