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**Abstract**

An extended investigation of the microstructure of A356 both as cast and processed by Liquid Hot Isostatic Pressing (LHIP) was performed using TEM, SEM and X-ray diffraction techniques. Specimens of the alloy in both conditions were solutionised for 8h at 520°C and aged for 6h at 160°C. TEM investigations revealed no significant influence of the L.H.I.P. process on particle precipitation kinetics and morphology. Pores were investigated and characterised by means of light and electron microscopy and statistical techniques. L.H.I.P. treatment reduced the pores mean size by about 1/3, the volume fraction by one order of magnitude and the shrinkage voids in number and volume.

**Riassunto**

La lega A356 da getto, dopo trattamento di L.H.I.P. è stata studiata per mezzo di tecniche di microscopia elettronica; TEM, SEM e di diffrazione a raggi X. Il materiale è stato solubilizzato a 520°C per 8 ore e quindi invecchiato a 160°C per 6 ore. Le indagini al TEM non hanno mostrato alcuna influenza significativa del processo di L.H.I.P. sulla cinetica e sulla morfologia di precipitazione. La percentuale di micro pori prodotti da gas intrappolati durante il riempimento dello stampo è stata calcolata per mezzo di osservazioni al microscopio ottico ed elettronico in scansione (SEM). I risultati sono il frutto di elaborazioni statistiche dei dati ottenuti. La dimensione media dei pori, dopo L.H.I.P., raggiunge valori inferiori ad 1/3 rispetto al materiale non trattato, e la frazione in volume è ridotta di un ordine di grandezza, inoltre i vuoti da ritiro subiscono una drastica diminuzione in numero e volume.

## Introduction

In an effort to reduce the costs and the fuel consumption of transportation means, new manufacturing technologies and light-weight metals, such as aluminium and magnesium alloys, are being used with increasing frequency. In automotive applications, weight reduction and high-quality components, coupled with lower costs, are a must. New production processes aim at improving mechanical properties, especially tensile strength and fatigue behaviour. Moreover, castings suffer from voids due to gas entrapped ($H_2$, $O_2$, $N_2$) by the liquid alloy during turbulent filling of the die and to subsequent shrinkage during solidification. The different coefficients of thermal expansion of primary particles and matrix also induce voids at interfaces. Both shrinkage voids and gas inclusion pores impair strength and fatigue resistance, as they can easily coalesce to premature fracture. Hot Isostatic Pressing (H.I.P.) has successfully overcome the susceptibility to shrinkage porosity of highly performing cast materials [1]. Unfortunately, its complexity makes H.I.P. a technology suitable for a narrow range of applications. Recently, this process has been modified by using, instead of a gas, a liquid at elevated temperatures to reach high pressures in few seconds (L.H.I.P.). On this basis, Metal Casting Technology has successfully engineered the process and extended it to mass production by increasing the volume of the room available for the pressing process [2]. During the L.H.I.P. process, the combination of pressure and temperature is able to close the voids due to shrinkage and the pores due to gases entrapment (such as $H_2$ and $O_2$) through the dissolution of gaseous inclusions within the lattice [2,3,4]. The novel development of H.I.P. process has constituted the starting point for Teksid's experimentation on castings of aluminium alloys using a mixture of salts particularly suitable for its low melting point, low density and viscosity, operating at temperatures ranging between 410 and 535°C. The castings are preheated at the temperature of the liquid and then introduced in the salt bath chamber, whose pressure is maintained for about 200 seconds over 1000 bars. The final step consists of quenching the castings to make them suitable for the subsequent hardening heat treatments [2-7].

The present paper aims to investigate the effect of the L.H.I.P. process on the microstructural properties of an A356 Aluminium alloy. To this end, we compared the microstructure of specimens before and after L.H.I.P. treatment aged at the same conditions.

## EXPERIMENTAL PROCEDURES

The A356 aluminium alloy, with the chemical composition reported in Table I, was sand cast at 715°C in the form of cylindrical specimens. One set of specimens (hereafter A) was solutionised at 520°C for 8h, quenched and aged at 160°C for 6h; another set (hereafter B) was solutionised, subjected to L.H.I.P. and then aged.
Light (LM) and scanning electron microscopy (SEM) investigations were conducted on specimens polished and etched with a 0.5% solution of HF in methanol. For TEM, slices were mechanically ground to about 120 mm, then etched with a 25% $HNO_3$ solution in methanol to about 80 mm. Discs were punched and finally thinned by double-jetting with a solution of a 1/3 $HNO_3$ in methanol at -30°C and 18/20V.

TABLE 1. COMPOSITION OF THE A356 ALUMINIUM ALLOY

| Si | Fe | Cu | Mn | Mg | Zn | Ti | Al |
|---|---|---|---|---|---|---|---|
| 6.50-7.50 | 0.60 | 0.25 | 0.35 | 0.20-0.45 | 0.35 | 0.25 | bal. |

## RESULTS AND DISCUSSION

X-ray Energy Dispersive Spectrometry (EDS) investigations on SEM and Electron Diffraction Pattern (EDP) indexing on TEM were used to identify the hardening phases in specimens A and B. The microstructure of A and B aged specimens is shown in Figure 1a, b. The Si particles, which had an aspect ratio ranging between 1.2 and 2.2, appeared dark and typically spherical or slightly elongated. The $Fe_2Si_2Al_9$ particles appeared as light-grey needle-like and the $FeMg_3Si_6Al_8$ phase appeared as chinese script-like and darker grey particles (see also Figure 2). A statistical evaluation of the volume fraction and dimensional variation of all the phases present in A and B specimens was carried out on TEM micrographs. The results - summarised in Table II - show no significant differences in particle volume fraction. The difference observed regarded the mean equivalent diameter(1) of the Si hardening particles. The mean equivalent diameter difference between A and B has been found to be: $(d_A - d_B) / d_A = |(6.6 - 8.2) / 6.6| = 24\%$, which shows

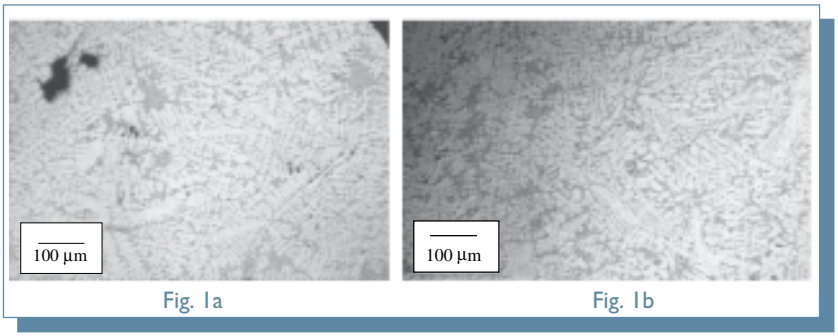


Fig. 1a Fig. 1b

Fig. 1: Light microscopic micrographs of the same specimen:
(a) solutionised at 520°C for 8h and then aged at 160°C for 6h,
(b) after L.H.I.P. under the same ageing conditions as (a).

(1) The equivalent diameter is a one-dimensional measure of the diameter of spherical shaped particles having the same volume of non-spherical ones

Fig. 2a

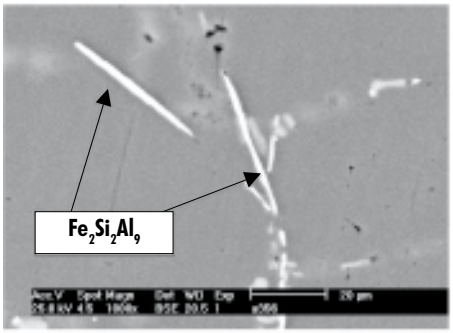


Fig. 2b

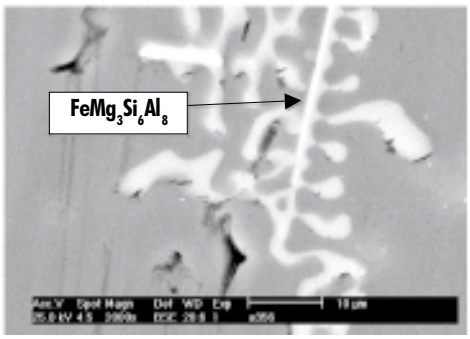


Fig. 2c

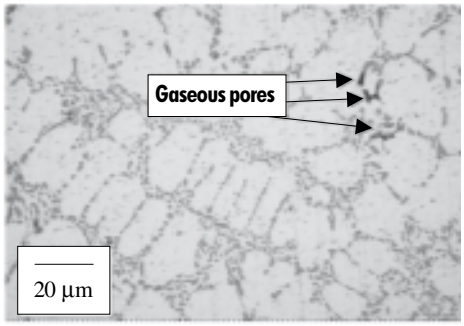


Fig. 2: Micrographs of second phases in A, (a): $Fe_2Si_2Al_9$, (b): $FeMg_3Si_6Al_8$,(c): all the present dark-grey rounded particles are Si

the B specimen's particles to be greater than in A.

The different phases, identified in specimens A and B, are shown in Figures 2 and 3. TEM investigations were also focused on the hardening microstructural precipitation. The hardness of the A and B samples was practically identical (92 ± 2 HB and 91 ± 2 HB, respectively), reflecting the lack of effectiveness of L.H.I.P. on hardening kinetics. The particles dimension of either A and B was bigger than GP and the relative difference did not affect significantly the hardness.

The LM micrographs of A (figure 1a) showed clearly a high concentration of pores and shrinkage voids, which were reduced by the L.H.I.P. process (figure 1b). The high pressures involved in the process produced a denser material by reducing micro and macroporosity. A detailed microstructural investigation allowed to identify the preferential location and the mechanism of microcracks formation. Figure 4 shows an intergranular region where the primary phases are located and this microstructural feature is common on A as well in B. In particular, the iron-rich particles ($Fe_2Si_2Al_9$ and $FeMg_3Si_6Al_8$) showed micro-voids and shrinkage cracks (the little dark striped zone) along the boundary with the matrix. Such a microstructural features at interfaces, under high stress concentration, can coalesce into cracks.

The presence of discontinuities at boundaries of the primary iron rich (>0.4wt.%) particles in the cast A356 alloy has been observed by Taylor and co-authors who analysed the role of the Si content [8]. They pointed out that the porosity at interfaces is minimised when solidification proceeded through ternary diagram Al-Si-$Fe_2Si_2Al_9$. In case of Si deficiency, the solidification proceeded thorough the binary Al-$Fe_2Si_2Al_9$ and the porosity volume fraction increased. An explanation of the primary particles on discontinuities at interfaces was the "restricted feeding theory", proposed by Mascré [9]. The theory basically suggests that $Fe_2Si_2Al_9$ and $FeMg_3Si_6Al_8$ intermetallic platelets formed within the interdendritic region during solidification and caused physical restrictions to the movement of the feeding liquid. It follows that voids are formed since interdendritic regions cannot be adequately fed.

The statistical evaluation of the porosity, on specimens A and B, has been focused on mean dimension (i.e. the equivalent diameter) and volume fraction. The results are reported in Table III and in Figure 5. The mean micro-pores dimension due to $H_2$ and $O_2$ [4] was up to 1/3 lower after L.H.I.P. and the porosity level (i.e. the micro-pores vol-

Table 2. Volume Fraction and Equivalent Diameter of all the detected second phases in specimens A and B. All the results have been processed by the dedicated software: Quantimet 500

| A356 (Cast) | Volume Fraction* (%) | Equivalent Diameter* (µm) | A356 (L.H.I.P.) | Volume Fraction* (%) | Equivalent Diameter* (µm) |
|---|---|---|---|---|---|
| Si | 11.80 | 6.6 | Si | 11.55 | 8.2 |
| $FeMg_3Si_6Al_8$ | 1.75 | 32 | $FeMg_3Si_6Al_8$ | 1.65 | 30 |
| $Fe_2Si_2Al_9$ | 1.95 | 32 | $Fe_2Si_2Al_9$ | 2.10 | 32 |

*: associated error within one standard deviation

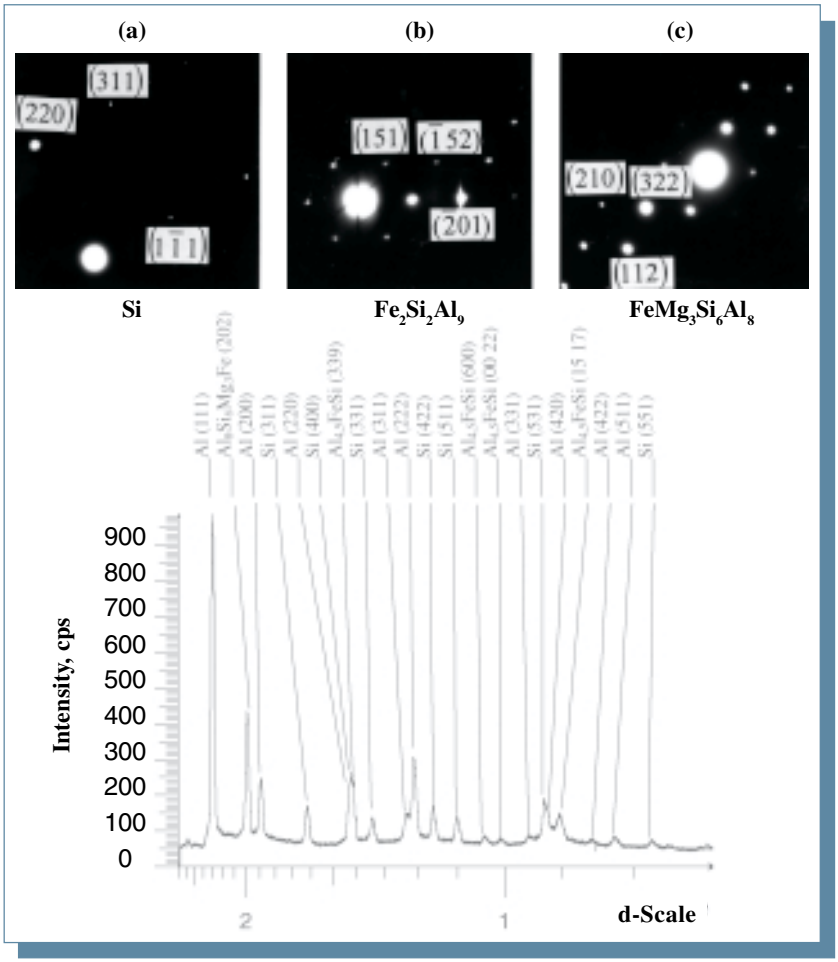


Fig. 3: X-ray diffraction analysis on the A356 aluminium alloy, and EDP of the different second phases detected, (a): Si, (b): $Fe_2Si_2Al_9$, (c): $FeMg_3Si_6Al_8$. The peaks of the cast and the L.H.I.P. materials practically overlap.

Table 3. Volume fraction and equivalent mean diameter of the cast and L.H.I.P. materials voids

| | Volume Fraction* (%) | Equivalent Diameter* (µm) |
|---|---|---|
| A356 - (A) | 2.8 | 7.0 |
| A356 L.H.I.P. - (B) | 0.6 | 2.7 |

*: associated error within one standard deviation

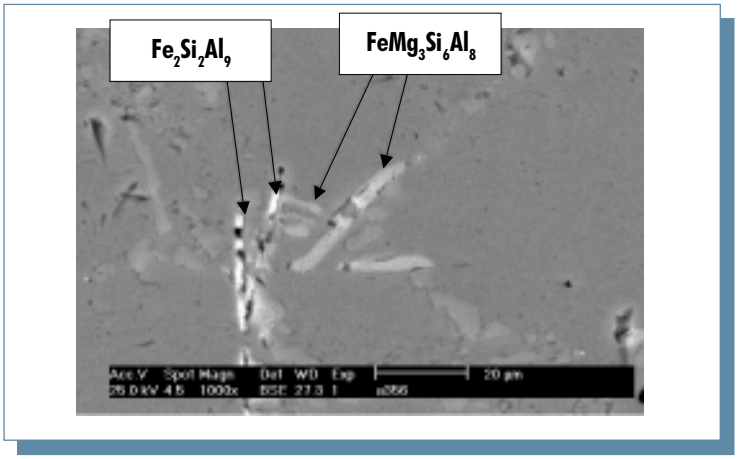


Fig. 4: Intergranular region where the two iron-rich second phases $Fe_2Si_2Al_9$ and $FeMg_3Si_6Al_8$ are located. A dark striped zone (a microcrack) appears along the boundary between the particle and the matrix

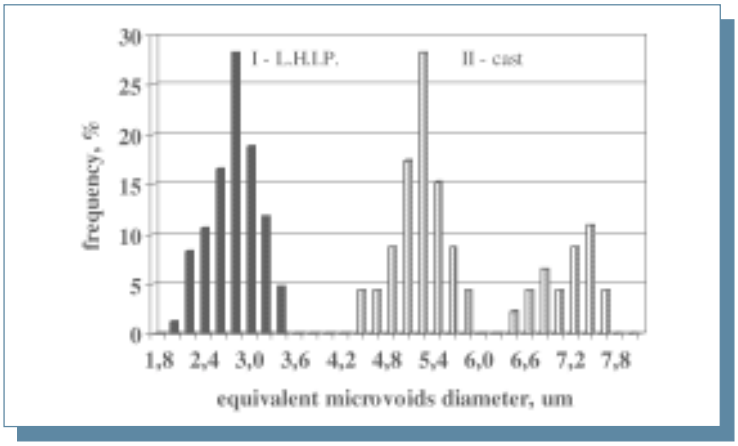


Fig. 5: Microvoid distribution. The cast and L.H.I.P. material are compared. The equivalent diameter is a one-dimensional measure of the magnitude of an object, and measures the diameter that the object would have if spherical (the equivalent particle volume is obtained by multiplying the square of the equivalent diameter by p/4). Left-side set of data: L.H.I.P. material; right-side data: cast specimens

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ume fraction) was down from 2.8% to 0.6% throughout the specimen volume, entailing a reduction in porosity by about 80%. The $N_2$ pores were not affected by L.H.I.P. [4]. Thus, the majority of macro pores (i.e. those having an equivalent diameter of the order of 0.7-0.6 mm) were reduced to micro pores of the order of 0.2-0.3 mm, and the former micro pores eventually disappeared.

The beneficial effect of the reduction in volume fraction of the mean pores size on tensile and fatigue behaviour produced by the L.H.I.P. process has been described in [4]. In particular, the fatigue properties of B specimens improved two-threefold compared with the A ones.

## CONCLUSIONS

The effect of LH.I.P. on the microstructure of aged samples of A356 was investigated by means of light and electron microscopy, and X-ray diffraction techniques. The results can be summarised as follow:

a) L.H.I.P. did not significantly influence the precipitation kinetics of Si particles that controlled the hardening of the alloy; the $Fe_2Si_2Al_9$ and $FeMg_3Si_6Al_8$ primary particles were stable at the temperatures of exposure.
b) L.H.I.P. dramatically reduced porosity from entrapped $H_2$ and $O_2$ in volume fraction and dimension. Morover, L.H.I.P. was capable of eliminating micro and macro voids.