Empirical models of mechanical behaviour of Al-Si-Mg cast alloys for high performance engine applications

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ABSTRACT
The main limitation of cast Al-Si-Mg alloys is the considerable influence of the solidification and heat treatment conditions on the final microstructure and consequently on the mechanical properties, that can thus widely vary in the same cast component. In this work we perform a deep microstructural and mechanical characterisation on specimens extracted from some A356 T6 gravity die cast cylinder heads. The aim of the work is to develop empirical models to successfully predict the local tensile properties of the casting from the main microstructural parameters and alloy hardness. As the former we considered and measured secondary dendrite arm spacing, shape and size of the eutectic silicon, grain size and percentage area of defects. The latter instead primarily depends on the heat treatment conditions. On the base of these data we propose a set of simple equations to predict the ultimate tensile strength and the proof strength with a mean error of 2% and the elongation to failure with a mean error of 20%. The equations allow the designer to know the local tensile behaviour without any tensile tests. Moreover, in a co-engineered design approach, the equations can link the post-processing results of the casting simulation software to the pre-processing phase of the structural software. This leads to less bench tests and to a great reduction of validation time of a new product.

RIASSUNTO
Il maggiore limite dei getti in lega Al-Si-Mg risiede nella elevata influenza delle condizioni di solidificazione e del trattamento termico sulla microstruttura finale e, di conseguenza, sulle proprietà meccaniche che possono dunque variare notevolmente nello stesso getto. In questo lavoro è stata condotta un’approfondita caratterizzazione microstrutturale e meccanica di provini ricavati da teste motore, in lega di alluminio A356 T6, ottenute mediante colata in conchiglia. L’obiettivo della ricerca è stato quello di mettere a punto modelli previsionali delle principali proprietà tensili della lega, sulla base dei principali parametri microstrutturali e dei valori di durezza. Fra i primi sono stati misurati: dimensione del grano, spaziatura tra i rami secondari dendritici (SDAS), forma e dimensione media del Si eutettico ed area percentuale dei difetti di solidificazione. La seconda dipende invece in prima misura dall’efficacia del trattamento termico. Sulla base di questi dati, vengono proposti modelli empirici per predire la resistenza a snervamento e trazione con un errore del 2% e l’allungamento a rottura con un errore medio del 20%. Le equazioni consentono al progettista la valutazione delle proprietà meccaniche locali del materiale senza bisogno di eseguire alcun test di trazione. Inoltre, nel caso di una progettazione di tipo integrato, tali equazioni possono rappresentare l’anello di giunzione tra le fasi di post-processing dei software di simulazione di processo e di pre-processing dei software di simulazione strutturale, portando ad una notevole riduzione dei test a banco e del tempo di validazione dei nuovi motori.

KEYWORDS
356, mechanical properties, microstructures, hardness, models.

Acronyms.  optical microscopy: OM; scanning electron microscopy: SEM; Transmission electron microscopy: TEM; Image analysis: IA; Grain size (d); Secondary dendrite arm spacing: SDAS; Aaspect ratio of eutectic silicon particles: AR; Area of eutectic silicon particles: A; Percentage area fraction of solidification defects: AF%; percentage cross section: CS% =100-AF%; Brinell hardness: HB; Ultimate tensile strength: UTS; 0.2% proof strength: YS; elongation to failure: E%;
INTRODUCTION

Excellent castability, corrosion resistance and high strength-to-weight ratio (which increases performance and fuel economy) have made cast Al-Si-Mg alloys suitable candidate materials for various applications in the automotive industry, such as engine blocks and cylinder heads. With these alloys (typically A356 and A357) it is possible to cast complex and thin-walled components through either sand, die and permanent mould casting with tensile strengths up to 350 MPa [1-3]. This high strength level is achieved through the T6 heat treatment. In particular Mg-Si precipitates provide strengthening through age hardening. The particular Mg–Si precipitates provide strengthening through age hardening. The static mechanical properties are mainly affected by SDAS and by the size and distribution of the second phase particles [9-13]. It is in fact widely reported that the tensile failure of Al-Si-Mg alloys occurs in three stages: (1) cracking of the eutectic silicon particle at low plastic strains (1–2%); (2) generation of localised shear bands with microcracks forming from the joining of adjacent cracked particles; (3) microcracks coalescence followed by propagation, leading to the final fracture. All these issues must be considered during the design of complex Al-Si-Mg cast components as a variety of local microstructures in the casting can lead to different mechanical properties. Most of the literature concerns the relationships between the solidification microstructure and mechanical properties as obtained by specimens produced under controlled laboratory conditions [4-12].

The cylinder heads we used in this study were produced through gravity die casting using the A356 aluminium alloy and were industrially heat treated at the T6 condition. In this work we first aimed at obtaining a map of the main microstructural parameters of the described cylinder head; second, we aimed at measuring the local mechanical properties using specimens directly machined from the cast; finally we found mathematical models to predict the local tensile properties of the casting knowing the main microstructural parameters and alloy hardness. The models allow the designer to predict the local tensile properties even in parts of the cast where the extraction of specimens is not possible. Moreover, in the case of a co-engineering design approach with intensive and reliable use of casting simulation software, the proposed predictive models can return the local mechanical properties of the cast that can be given as input to the structural simulation software. This leads to less bench tests and to a great reduction of validation time of a new product.

EXPERIMENTAL

THE CYLINDER HEAD

A primary A356 aluminium alloy (EN AC 42100) is used to fabricate cylinder heads (Fig. 1a) through gravity die casting. The chemical composition of the alloy, as obtained by spectrometric analyses, is listed in Tab. 1.

The A356 ingots are first remelted in a gas furnace. The hydrogen level in the melt is reduced by using a rotary lance degasser and a high purity argon gas. Grain refinement and eutectic Si modification are achieved by adding, during the degassing process, commercial Al-Ti-B (Al-5%Ti-1%B) and Al-10wt.%Sr master alloys, respectively. The level of hydrogen is controlled by verifying that, after the degassing operation, density don’t exceed the lower limit of 2.64 Kg/dm³. Chemical composition of the melt was controlled through spectrometric analyses. The melt is then poured into a maintenance furnace at 730°C and then poured into the mould. Solidification of the cast occurs in about 20s since the end of the filling. After 9 minutes, the die is opened. The first step of the sand handling consists of high temperature exposure (500°C) and shakeout operations. The casting system is then removed and the cast is brought to the T6 condition through solution (535°C for 4.5h), water quenching (20-60°C) and aging (165°C for 4.5h). The time between solution and aging treatments (“pre-aging”) during which castings remain at room temperature can range between 0 and 2h. If any problem in the managing of the furnaces occurs so that a longer pre-aging time might be reached, the castings are stored at –30°C. Brinell hardness tests are carried out on the whole production, on fixed points of the castings to verify the effect of the heat treatment. Target values ranging between 95 and 105 HB. The experimental analysis was carried out by extracting samples and specimens from three cylinder heads in the T6 conditions and one in the as-cast conditions.

MECHANICAL TESTING

50 specimens for mechanical testing were cut from different parts (Fig. 1b) of the cylinder head in the as-cast condition. Before the machining of the samples, the previously mentioned heat treatment was performed. Different set of specimens were obtained, one for each different pre-aging time (between 0 and 2h). Mechanical characterisation of the heat treated specimens was performed through (1)

Table 1. Chemical composition of the A356 Al alloy (weight %)

<table>
<thead>
<tr>
<th></th>
<th>Si</th>
<th>Mg</th>
<th>Fe</th>
<th>Cu</th>
<th>Mn</th>
<th>Zn</th>
<th>Ti</th>
<th>Sr</th>
<th>B</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>7.24</td>
<td>0.42</td>
<td>0.138</td>
<td>&lt;0.001</td>
<td>0.007</td>
<td>0.003</td>
<td>0.120</td>
<td>0.015</td>
<td>0.0354</td>
<td>Bal.</td>
</tr>
</tbody>
</table>
Brinell hardness tests (HBW 2.5/62.5) according to the ASTM E 10–08 standard [14] and (2) tensile tests according to the UNI EN 10002-1:2004 standard [15]. Tensile tests were performed on a screw testing machine at room temperature with a nominal strain rate of 3.3·10⁻⁴ s⁻¹, and using 50 flat specimens (Fig. 1c) with a gauge length L₀=30mm and a cross-sectional area S₀=25mm². We tested 6 specimens for each pre-aging condition to evaluate the 0.2% proof strength, the ultimate tensile strength and the elongation to failure.

MICROSTRUCTURAL ANALYSIS

We performed OM microstructural analysis on sections of the heat treated cylinder head (Fig. 1d) and on a cross section near to the fracture surface of the tensile specimens. Metallographic specimens were prepared using standard metallographic techniques including mechanical grinding and polishing with 9 µm and 1 µm diamond paste (according to the ASTM E3 [16]). We revealed the grain structure using electrolytic etching with Barker’s reagent (5ml HBF₄ (50%wt.), 100ml distilled water) and anodising for 80s at 15V (according to ASTM E 883 standard [17]). We performed IA in order to measure grain size, SDAS, aspect ratio and area of the eutectic silicon particles. We evaluated these microstructural parameters by considering about 30 optical micrographs for each tensile specimen and about 2000 micrographs for each analysed section of the casting. Such a high number of micrographs was necessary due to the wide surface (about 1 dm²) of the sections. The grain size was measured on the electrolytically etched samples observed under polarised light, according to the Heyn Linear Intercept Procedure described in the ASTM E112 standard [18]. We measured the value of SDAS by identifying and measuring aligned groups of secondary cells. The value of SDAS was then calculated as SDAS=L/n, where L is the length of the line drawn from edge to edge of the measured cells and n the number of the dendrite cells. At least 100 cells for each sample were considered. Aspect ratio and area of the eutectic Si particles was evaluated as the ratio of major and minor axes of the equivalent ellipse on about 2000 particles for each sample. This number of particle was that one identified as consistent by some previous analysys. It was obtained by considering about 20 high magnification micrographs for each specimen (corresponding to about 1% of its cross section).

We used a Zeiss Evo®50 scanning electron microscope to analyse the fracture surfaces of the tensile specimens. The percentage area fraction of the solidification defects (such as gas pores and cavity shrinkages) on the fracture surfaces was evaluated by image analyses. The actual cross section of the tensile specimens was estimated as CS% = 100 – AF%. For this evaluation, the total area of defects measured on the fracture surface was divided by the nominal cross section of the specimen (measured with a micrometer before the tensile test). This choice can be explained by considering that: 1) IA on a single random metallographic section of the specimen cannot give a true evaluation of the defects content; 2) it is highly probable that the fracture surface of the specimen is the one with the highest defects content. IA of OM and SEM micrographs was performed with the Image pro-Plus® software.

CORRELATION ANALYSIS

We first used a correlation matrix [19] to define the correlations among mechanical properties and microstructural parameters. Given a nxn correlation matrix A, where n is the total number of considered mechanical properties and microstructural parameters, each element A[i,j] represents the Pearson’s correlation coefficient between property/parameter i and property/parameter j (in the range [-1,1]). In particular the higher is the absolute value of the coefficient, the higher the correlation between the properties/parameters. Once the parameters with highest correlation with the mechanical properties were identified, their relationships with the mechanical properties were evaluated using multiple variable regression analysis (performed with the XLStat® software).

RESULTS AND DISCUSSION

MECHANICAL TESTING

Hardness tests on the tensile specimens have shown the effect of pre-aging on the alloy hardness after the T6 heat treatment. Tab. 2 shows the results of the tests. As also reported in [20], it is clear how longer pre-aging time results into a less hard final alloy. Considering the case where no pre-aging occurs as ideal situation, in our experiments the hardness loss was about 15 HB for pre-aging time of 60 min. Pre-aging time longer than 60 min., on the other hand, did not have any further significant effect on the alloy hardness. The results of the tensile tests showed that the pre-aging time variation led to a similar effect on the 0.2% proof strength and ultimate tensile strength. A decrease of about 50 MPa occurs with 60 min. of pre-aging time for both the tensile properties. On the other hand, the maximum values of elongation to failure of the different sets of specimens seem to increase with the pre-aging time. The effect of the pre-aging on the minimum values of elongation to failure seems instead negligible. Considering both the minimum and maximum values of E%, it seems clear that the higher is the pre-aging time, the larger the scatter in the data. By increasing the pre-aging time, we obtained a great reduction in the alloy hardness but a non systematic elongation to failure increase. The hardness (pre-aging time) could not so be considered the unique parameter we have to consider in the...
The microstructure of the alloy is typical of a hypoeutectic Al-Si alloy, with primary α-Al dendrites and eutectic Si particles distributed around the Al dendrites to form a cell pattern periodically repeated across the metallographic surface. However, as can be seen from the micrographs reported in Fig. 2-4 and from the digital IA measurements reported in Tab. 3, significant microstructural differences were observed between the samples extracted from different zones of the castings. The SDAS ranged between a minimum value of about 25 µm (Fig. 2a) and a maximum value of about 70 µm (Fig. 2b) depending on the local solidification rate. In fact, as reported in [6], SDAS [µm], in the A356 alloy, can be computed as:

\[ \text{SDAS} = 39.4 R^{0.317} \]  

(1)

where \( R = \frac{dT}{dt} \) [°C/s] is the mean cooling rate during solidification of the α-Al phase. High solidification rates, reached for example near the combustion chamber (Zone 2 in Fig. 1b), reduce SDAS and thus generally lead to an improvement of almost all the mechanical properties (yield and tensile strength, as well as elongation to failure and fatigue resistance) [9, 10, 12]. Higher values of SDAS were instead measured in the camshaft seat zones (Zone 1 in Fig. 1b) where a large and complex system of cores and feeders is present. The grain size ranged between a minimum value of about 200 µm to a maximum of 500 µm. Representative optical micrographs of the samples, electrolytically etched and observed under polarised light, are shown in Fig. 3. The grains surrounded by the eutectic Si (dark in the micrographs) can be clearly seen. Due to the efficiency of Ti-B as a nucleation agent, a homogeneous distribution of the grain size in the casting was observed, with a weak relationship with the cooling rate. Since they control the mechanisms of failure, size, morphology and distribution of the eutectic silicon particles are also important microstructural parameters in cast Al-Si alloys [9-13]. It is well known that unmodified eutectic silicon has an undesired needle-like morphology that acts as a stress concentrator, reducing strength and ductility. The eutectic silicon particles can be modified to obtain a rounded morphology through rapid solidification, chemical modification and thermal modification in the solid state [21-24].

During the industrial production of these cylinder heads, a target value of 150 ppm (weight %) of Sr is introduced into the melt to obtain a finer and rounded eutectic silicon. The microstructural analyses, carried out on specimens cut from different zones of the cast, showed a different degree of eutectic Si modification partially depending on the cooling rate. As reported in [12, 22, 23], by increasing the solidification rate, the size and the aspect ratio of the eutectic silicon particles decrease (Fig. 4a). On the contrary, in zones where the cooling rates are lower, the eutectic Si particles appear larger and less modified, with a more lamellar morphology (Fig. 4b).

We measured defects content (gas pores and shrinkage cavities) using SEM on fracture surface of the tensile specimens (Fig. 5a) and using OM on some section of the casting (Fig. 1d). The results of the IA on the fracture surfaces (Tab. 3 and Fig. 5b) highlighted the very low level of porosity in this complex cast component. The percentage area fraction of the casting defects ranged from 0% to 4.2% for all the specimens except for one (extracted from a peripheral zone of the casting) where the presence of a cold-shut (Fig. 5a) and a total

| Tab. 2 Results of the tensile and hardness tests carried out on specimens extracted from two cylinder heads and subjected to different pre-aging times. The last raw (reporting min and max values for all the tested specimens) shows the great variation of properties achievable from two nominally identical castings.

<table>
<thead>
<tr>
<th>pre-aging, min</th>
<th>HB min</th>
<th>HB max</th>
<th>YS, MPa min</th>
<th>YS, MPa max</th>
<th>UTS, MPa min</th>
<th>UTS, MPa max</th>
<th>E% min</th>
<th>E% max</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>111</td>
<td>116</td>
<td>254</td>
<td>266</td>
<td>297</td>
<td>333</td>
<td>1.5</td>
<td>9.3</td>
</tr>
<tr>
<td>10</td>
<td>103</td>
<td>114</td>
<td>227</td>
<td>266</td>
<td>285</td>
<td>325</td>
<td>3.9</td>
<td>8.4</td>
</tr>
<tr>
<td>20</td>
<td>106</td>
<td>110</td>
<td>234</td>
<td>259</td>
<td>272</td>
<td>328</td>
<td>1.5</td>
<td>12.9</td>
</tr>
<tr>
<td>30</td>
<td>100</td>
<td>111</td>
<td>216</td>
<td>256</td>
<td>262</td>
<td>315</td>
<td>2.2</td>
<td>5.6</td>
</tr>
<tr>
<td>45</td>
<td>95</td>
<td>103</td>
<td>199</td>
<td>223</td>
<td>252</td>
<td>289</td>
<td>2.6</td>
<td>8.2</td>
</tr>
<tr>
<td>60</td>
<td>96</td>
<td>101</td>
<td>203</td>
<td>214</td>
<td>248</td>
<td>296</td>
<td>1.9</td>
<td>13.2</td>
</tr>
<tr>
<td>120</td>
<td>93</td>
<td>103</td>
<td>193</td>
<td>266</td>
<td>243</td>
<td>295</td>
<td>1.9</td>
<td>14.2</td>
</tr>
<tr>
<td>Total range:</td>
<td>93</td>
<td>116</td>
<td>193</td>
<td>266</td>
<td>243</td>
<td>333</td>
<td>1.5</td>
<td>14.2</td>
</tr>
</tbody>
</table>

| Tab. 3 Results of the IA measurements carried out on the metallographic specimens and on the fracture surface of the 50 tensile specimens extracted from the cylinder head.

<table>
<thead>
<tr>
<th></th>
<th>Min</th>
<th>Max</th>
<th>Average</th>
<th>St. Dev.</th>
</tr>
</thead>
<tbody>
<tr>
<td>SDAS, µm</td>
<td>25</td>
<td>70</td>
<td>50</td>
<td>13</td>
</tr>
<tr>
<td>d, µm</td>
<td>205</td>
<td>501</td>
<td>339</td>
<td>66</td>
</tr>
<tr>
<td>A, µm²</td>
<td>8</td>
<td>19</td>
<td>13</td>
<td>2</td>
</tr>
<tr>
<td>AR</td>
<td>1.04</td>
<td>1.80</td>
<td>1.53</td>
<td>0.11</td>
</tr>
<tr>
<td>CS%</td>
<td>88.75</td>
<td>100</td>
<td>98</td>
<td>1.84</td>
</tr>
</tbody>
</table>
amount of defect of about 12.25% were observed.

IA on the four sections analysed using OM showed a maximum value of percentage area fraction of defects of about 2%, confirming the initial hypothesis that in a tensile specimen the fracture surface is the one with the highest content of defects.

The extremely poor content of defects measured using both OM and SEM analysis, indicated the high quality of the design of the bottom-gated and top-fed running system of this extremely complex casting.

**CORRELATION ANALYSIS**

Tab. 4 depicts the previously described correlation matrix. Note that only absolute values of the Pearson’s coefficient were considered relevant in our analysis. YS is not significantly affected by the solidification microstructure while it is just depending on the alloy hardness. A hardness increase from 95 to 115 HB leads to a corresponding increase of the YS from about 200 to 260 MPa. As widely reported [25-27], the relationship between hardness and 0.2% proof strength of heat treatable aluminium alloys is mainly related to the strengthening effect of the Mg2Si precipitates induced by the T6 heat treatment. This kind of precipitates are in fact finely dispersed in the α-Al matrix, hindering the dislocations motion and therefore limiting the plastic deformation of the alloy. In this mechanism, microstructural parameters as SDAS play a minor role, for example promoting or inhibiting the diffusion of strengthening alloying elements during solution. A linear single variable (hardness) equation for 0.2% proof strength prediction was so chosen:

\[ YS = 3.419 \times HB - 127.6 \quad R^2 = 0.910 \quad (2) \]

The average error between the predicted YS values and the experimental ones (Fig. 6) was only 2.3% (about 5 MPa), while the maximum error was about 7%.

Tab. 4 also shows that, unlike the YS, the UTS and the E% were found to depend on the solidification microstructure. Hardness, SDAS, percentage cross section and area of eutectic silicon particle are the most effective parameters on UTS. As reported in [10], these microstructural parameters can influence the mechanical behaviour of the material. Crack nucleation and propagation occur in fact at discontinuities of the matrix (such as solidification defects and eutectic silicon particles) and are faster the larger the size of these discontinuities is. By considering the data reported in Tab. 2, we measured the maximum values of UTS for low SDAS values. A decrease of SDAS from 55 µm to 35 µm leads to an increase of UTS of about 30 MPa. The area of eutectic silicon and area fraction of defects registered a similar behavior: the higher the parameter the lower the property.

Based on the previously reported findings, an empirical equation (3) to predict the UTS taking into account hardness (HB), secondary dendrite arm spacing (SDAS) and percentage cross section (CS%) of tensile specimen was proposed:

\[ UTS = 0.183 - CS\% - 0.803 \times SDAS - 0.121 + HB^{0.888} \quad R^2 = 0.899 \quad (3) \]

The average error between the predicted UTS values and the experimental ones (Fig. 7) was only 2.1% (about 5 MPa), while the maximum error was about 6%. From a practical point of view this model easily allows us to know the local UTS of the material also where tensile specimen cannot be extracted from the casting. Moreover, since hardness, SDAS and solidification defect content can be predicted by casting simulation software [28-30], it seems clear how this model could be applied in a co-engineered design approach.

Referring to equation (3), since solidification defects act as stress raisers (leading to an earlier fatigue crack nucleation) and higher hardness values are related to a more effective age hardening, positive exponents are needed for both percentage cross section and Brinell hardness. On the contrary, for SDAS, a negative exponent is needed, since a finer microstructure generally leads to higher mechanical properties [9-13].

The same considerations can also be made for the predictive equation of elongation to failure, reported below:
E% = 5074.8 * CS%2.951 * SDAS1.239 * HB-2.857 * A-0.957
R² = 0.813

(4)

It is worth noting that, despite the deep metallographic and fractographic analyses carried out in this study, the proposed model error when predicting the elongation to failure is significant (Fig. 8). A 20% average error between the predicted E% values and the experimental ones was in fact measured with peaks of about 80%. This large scatter between measured and calculated values is probably due to morphology and location of the solidification defects in the tensile specimen or to the size and distribution of Mg2Si strengthening precipitates. Analysis on the latter should be carried out using TEM. A deeper analysis can be performed to evaluate the influence of these parameters on the elongation to failure. However, the impossibility to determine them through casting simulation software has led us to neglect them in the present study.

On the contrary, the input variables of equation (4) can be simply evaluated with direct measurements or via simulation, except for the mean value of eutectic silicon area (A). This parameter ranges between 8 µm² and 16 µm² in the entire casting and is related with SDAS. Finer values of SDAS (<45µm) lead to a mean value of about 10 µm² while larger values of SDAS (>45µm) lead to a mean value of 14 µm². This behaviour suggests that we could use equation (4) introducing pre-defined values of A according to the SDAS distribution in the casting.

VALIDATION AND APPLICATION OF THE MODELS

To verify the predictive models efficacy, we extracted six specimens from two different batch production cylinder heads. All the input variables of the empirical models for the six specimens have been evaluated (Tab. 5) and the tensile tests were performed. Predicted and experimental values of the mechanical properties of the six specimens are reported in Fig. 9.

The data reported in Fig. 9 suggest that the predicted values are sufficiently close to the experimental one. The empirical models give the 0.2% proof strength and ultimate tensile strength of the alloy with a maximum error (underestimation) of 6%. On the contrary, the predicted elongation to failure leads to overestimate the actual elongation to failure of the alloy of an average value of 40%. It is also worth noting that, despite the percentage error, the predicted values follow the increasing/decreasing patterns of the mechanical properties.

In order to clearly highlight the potential of the models, especially when used in combination with simulation software that can return good prediction of SDAS, defects content and hardness [28-30], we realised a map (Fig. 10) of the mechanical properties of the casting, by using the microstructural parameter measured on a section of the cylinder head (Fig. 1d).

CONCLUSIONS

In this work we propose empirical equations to predict tensile behaviour of A356 T6 gravity die casting as a function of hardness and microstructural parameters.
Q 0.2% proof strength can be predicted as a linear function of the hardness alloy. The mean error in the property prediction on the specimens extracted from industrial castings was 4% of the actual measured value.

Q The ultimate tensile strength of the alloy was found to depend on SDAS, defects content and hardness. The mean error in the property prediction on the specimens extracted from industrial castings was 3% of the actual measured value.

Q The elongation to failure of the alloy was found to depend on SDAS, defects content, hardness and eutectic silicon size. The mean error in the property prediction on the specimens extracted from industrial castings was 40% (overestimation) of the actual measured value.

Q The shape of eutectic silicon and the grain size were not important parameter for the prediction of any of the tensile properties. The high error in the prediction of elongation to failure of the alloy might be related to the variable that were not considered in this study such as position and shape of casting defects, size and distribution of strengthening precipitates (Mg2Si).

Q The good results obtained suggest that the proposed model could be applied to the modern casting simulation software to obtain a mesh/map of mechanical properties that could be given as input to structural simulation software just during the component design phase.

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Constitutive and stochastic models to predict the effect of casting defects on the mechanical properties of High Pressure Die Cast AlSi9Cu3(Fe) alloys

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ABSTRACT
The effect of casting defects on mechanical properties of a high-pressure die-cast AlSi9Cu3(Fe) alloy is reported. A series of U-shaped structural components are cast using a combination of injection parameters and pouring temperatures in order to generate different types and amount of casting defects throughout the casting. It has been found that castings contain defects, primarily pores and oxides, and that the presence and distribution of these defects are highly sensitive to the process conditions. Moreover, significant variations of the defect distribution have, however, been found in castings produced under the same conditions, indicating the stochastic nature of defects in die castings. The tensile properties are affected by the amount and distribution of defects and are determined by the defect area fraction. The influence of casting defects on mechanical properties are investigated through a theoretical verification based on constitutive and stochastic models. The analytical approach, based on the Ghosh constitutive model of tension instability, correctly indicates the trends of the experimental results, while the Weibull statistics evidences how the scale parameter and the Weibull modulus are strongly affected by the casting conditions. An integrated stochastic-analytical approach is then proposed and it appears to be applicable to describe the tensile properties in terms of fractographic defects and cumulative failure probability $P_i$.

RIASSUNTO
Nel presente lavoro è stato studiato l’effetto dei difetti sulle proprietà meccaniche di una lega d’alluminio AlSi9Cu3(Fe) pressocolata. È stata realizzata una serie di componenti strutturali con profilo ad U utilizzando una combinazione sistematica dei parametri di iniezione e della temperatura di colata, al fine di generare diversi tipi e quantità di difetti all’interno dei getti. Si è quindi riscontrato che i getti pressocolati contenevano difetti, principalmente porosità e ossidi, e la distribuzione di questi fosse sensibilmente influenzata dalle condizioni di processo utilizzate. Si sono evidenziate, tuttavia, variazioni significative nella distribuzione dei difetti nei componenti realizzati con le medesime condizioni di processo, indicando quindi come i difetti presentino una natura stocastica nei componenti pressocolati. Le proprietà meccaniche sono risultate influenzate dalla quantità e dalla distribuzione dei difetti, e determinate dalla frazione d’area occupata dai difetti stessi sulla superficie di frattura delle provette di trazione. L’influenza dei difetti sulle proprietà meccaniche è stata studiata anche attraverso un approccio teorico basato sull’applicazione di alcuni modelli costitutivi e stocastici. L’approccio analitico, basato sul modello costitutivo di Ghosh, ha indicato correttamente la tendenza dei risultati sperimentali; contemporaneamente il modello statistico di Weibull ha evidenziato come il parametro di scala e il modulo di Weibull sono influenzati sensibilmente dalle condizioni di pressocolata. Un approccio integrato stocastico-analitico è stato quindi proposto e si è dimostrato applicabile per descrivere le proprietà meccaniche in termini di difetti contenuti sulla superficie di frattura e probabilità di rottura $P_i$.

KEYWORDS
Aluminium alloys; Defects; Oxide films; Porosity; Mechanical properties; Modelling; High pressure die casting.
INTRODUCTION

The unfailing increased use of light alloys in the automotive industry is, above all, due to the need of decreasing vehicle’s weight. The same need has to be taken into account in order to face up also both energetic and environmental requirements [1]. In terms of application rates, Al and its alloys have an advantage over other light materials, such as Mg and Ti alloys. The reduced prices, the recyclability, the development of new improved alloys, the increased understanding of design criteria and life prediction for stressed components and an excellent compromise between mechanical performances and lightness are the key factors for the increasing demand of Al alloys. A great contribution to the use of Al alloys comes from improvements in casting processes, which allow to increase the production, to reduce the cycle time, and to manufacture complex-shaped castings with thin wall thickness. Among the recent casting techniques, the high-pressure die-casting (HPDC) is largely used by the automotive sector since fulfils the above advantages [1,2].

A limit to large diffusion of HPDC remains the final quality of castings. While the combination of high speed casting and high cooling rate gives the possibility of thin walled castings, the associated turbulence remains the major source of inner and surface casting defects, which have a deleterious effects on mechanical properties. In HPDC if a number of parameters is not adequately determined and adjusted, the quality of the die cast part results rather poor [3,4]. Macro segregation of eutectic, primary intermetallic particles [5,6] and α-Al crystals [7], porosity, oxide bifilms and confluence welds [8] are addressed as typical HPDC defects.

By means of casting parameters’ adjustments, foundrymen try to restrict and isolate the major part of defects into regions of the casting which are not mechanically stressed during normal working. Further, thin-walled castings, like those produced by HPDC, are more affected by the presence of defects since a single macrodefect can cover a significant fraction of the cross-section area. A number of researchers has investigated the influence of casting defects on the mechanical properties both of gravity cast [9-13] and high pressure die cast aluminium alloys [14-16]. However, the works of Gokhale et al. [17] and Timelli et al. [16] evidenced how the mechanical properties decrease monotonically with increasing the area fraction of defects revealed on the fracture surfaces both of gravity and high pressure die cast aluminium specimens. The common conclusion was that even high integrity castings contain defects and thus it is important to predict their effect on final mechanical properties.

In literature, several approaches based on through-process modelling for prediction of the structural behaviour of HPDC magnesium and aluminium alloys components subjected to static and dynamic loads have been suggested [14, 16, 18-20]. Generally, two different routes based on constitutive models [21-23], or statistical and stochastic approaches [11, 24-27] are used. Cáceres [21] and Lee [23] reported that the theoretical approach based on the Ghosh constitutive model [28] can accurately predict the experimental tensile properties of aluminium alloys, even though they used a simple constitutive model. In the model, based upon the tensile instability, the tensile strength and deformation of material with internal discontinuities significantly depend upon the fraction of internal discontinuity, the strain rate sensitivity and strain-hardening ability.

On the other side, the effect of structural defects on mechanical properties have been characterized by Weibull statistics, more specifically, by the two-parameter Weibull modulus [11, 26, 29]. In these early studies, the Weibull modulus appeared to be a useful measure of the reliability of the casting process. Since then, the two-parameter Weibull modulus has been extensively used to characterize the tensile properties, especially the tensile strength. Recently, the use of three-parameter Weibull statistics has been explored to illustrate its superior analytical potential over the traditional two-parameter approach [30]. The three-parameter Weibull analysis provides new information. In particular, minimum values of strength below which the material is extremely unlikely to fail are found.

In the study, the influence of casting defects on mechanical properties of secondary AlSi9Cu3(Fe) die cast alloy was investigated through a systematic experimental approach, with a theoretical verification based on constitutive and stochastic models. Moreover, a combination of injection parameters and pouring temperatures were chosen in order to generate different types and amount of defects throughout the casting.

THEORETICAL ASPECTS

CONSTITUTIVE MODEL OF TENSION INSTABILITY

When porosity or an equivalent defect is present in a tensile specimen, the load bearing area is reduced. Thus, the defective region will yield first, concentrating the strain. The rate of strain concentration can be calculated considering the strain hardening ability of the material. A geometric defect that locally reduces the load bearing area of a tensile specimen results in the formation of an incipient neck. The growth of this neck can be described using the Ghosh constitutive model for the development of plastic instabilities [28]. If the neck is not sharp or the strains involved are not large, it may be considered that only one significant stress exists in either the uniform section or the local inhomogeneity. Under the assumption that the material containing internal discontinuities experiences a tensile load under axial local equilibrium and the effects of strain rate can be neglected, the conventional equation for stress distribution can be expressed in terms of load carrying area as [21, 22, 28]

\[ \sigma_i (1 - f) A_0 e^{\varepsilon_i} = \sigma_0 A_0 e^{\varepsilon_c} \]  

(1)

where \( \sigma_0, \varepsilon_c \) and \( \sigma_i, \varepsilon_i \) are the true stresses and strains in and outside the defect region, respectively, \( A_0 \) is the initial cross section of the specimen and \( f \) is the area fraction covered by defects.

A numerical solution of eq. (1) can be obtained by means of the following Hollomon constitutive equation [31]
where $\sigma$ and $\varepsilon$ are the true stress and plastic strain, respectively, while $K$ is the alloy’s strength coefficient and $n$ the strain hardening exponent. The substitution of eq. (2) into (1) leads to [21, 22, 28]

$$1 - f e^{\sigma_n/n} = e^{\varepsilon_n}$$

which relates the strain inside the defect region $\varepsilon_i$ to the strain outside $\varepsilon_s$.

Moreover, since the true uniform strain of sound material is equivalent to the strain hardening exponent under maximum loading conditions ($\varepsilon_s = \varepsilon = n$), the tensile stress $\sigma_i^*$, with a defect content $f$, can be expressed as the following equation from the power law of eq. (2) [21, 22, 28]

$$\frac{\sigma_i^*}{\sigma_s} = \left(\frac{\varepsilon_i}{\varepsilon_s}\right)^n$$

where $\sigma_s$ and $\varepsilon_s$ are the maximum true stress and maximum strain to fracture of sound material, respectively, and $\varepsilon_i$ is the premature true strain of material which has a defect content $f$.

Therefore, the predictions of this model depend on the values of $n$ and $f$, i.e. for a given strain hardening exponent, only the area fraction of defects revealed on the fracture surfaces is important.

**WEIBULL STATISTICS**

Structural defects such as porosity and oxide inclusions or microstructural features such as Si eutectic or intermetallic phases constitute the initiation point of fracture, since they lead to the largest stress concentration. This point will constitute the weakest link and when the mechanical properties of a group of nominally similar specimens is measured, the data acquired are usually scattered. The statistical distribution that can reasonably model such a distribution was proposed by Weibull [32] and was originally used to analyse the yield strength and fatigue behaviour of steel [33]. The common Weibull distribution function is expressed as

$$P_i = 1 - \exp\left[-\left(-\frac{\chi}{\eta}\right)^{\beta}\right]$$

where $P_i$ is the cumulative fraction of specimen failures (in tensile test); $\chi$ is the variable being measured (ultimate tensile strength or elongation to fracture); $\lambda$ is the threshold parameter, i.e. the characteristic stress (or strain) below which no specimen is expected to fail; $\eta$ is the scale parameter, i.e. the characteristic stress (or strain) at which 63.21% of the specimens has failed; $\beta$ is the shape parameter, alternatively referred to as the Weibull modulus [11,30,32,33]. This configuration of the Weibull distribution is the three-parameter configuration. Generally, for aluminium castings, the two-parameter form is widely adopted and can be expressed with the threshold value $\lambda$ taken as zero.

The Weibull distribution is asymmetrical about the mean strength (or strain) if compared to Gauss distribution. Simplifying eq. (5) by assuming $\lambda$ as zero, the Weibull distribution can be converted into the linear form

$$\ln[\ln(1/(1 - P_i))] = \beta\ln(\chi) - \beta\ln(n)$$

where $i$ is the ranked position of the specimen strength (or strain) in that set of castings and $n$ is the total number of specimens.

**EXPERIMENTAL PROCEDURE**

The secondary AlSi9Cu3(Fe) cast alloy (EN AC-46000, equivalent to the US designation A380) was supplied as commercial ingots, which were melted in a 500 kg SiC crucible in an electric resistance furnace. Before pouring the melt is held in the furnace at 690 ± 5°C for 1 h to ensure homogeneity and dissolution of the present intermetallic. Periodically, the molten metal was manually skimmed and stirred with a coated paddle to avoid any type of sedimentation. The furnace temperature is the holding temperature commonly used for EN AC-46000 type alloys, which is enough to avoid sludge formation [34,35]. The chemical composition, measured on separately poured samples, is shown in Table 1.

For R&D purposes, a die for U-shaped casting was made. The CAD model of Al casting with runners, gating and overflow system is displayed in Figure 1. The U-shaped casting with 2.5 mm thickness was coupled with ribs, with ~5 mm thickness, which are generally locations of high defect content. The castings were produced in a Müller-Weingarten cold chamber die-casting machine with a locking force of 7.4 MN. The weight of the Al alloy casting was 3.3 kg, including the runners, gating and overflow system. A detailed description of the HPDC machine, the casting procedure, and the process parameters is given elsewhere [16]. Briefly, 10 to 15 castings were scrapped after the start up, to reach a quasi-steady-state temperature in the shot chamber and die. Oil circulation channels in the die served to stabilize the temperature (at ~230°C). The fill fraction of the shot chamber, with a 70 mm inner diameter, was kept at 0.56. A combination of injection parameters and pouring temperatures were chosen in order to generate different types and amount of casting defects. Table 2 summarizes the
In the P1 reference process, the plunger speed was kept constant in the first phase and a rapid acceleration was applied in the second phase, i.e. at the beginning of die filling. The same profile was used for T2 process but the pouring temperature was 50°C lower. In the other shot profiles the plunger was slightly higher in the first phase, minimizing however the air entrapment in the slow shot. The main differences regarded the variations of the switch point between the first and second phase: the commutation point was anticipated in the P3 process and postponed in the P2 and P4 processes. Further, the plunger velocity in the fast shot was reduced in the P3 and P4 profiles. By means of a dynamic shot control system in the HPDC machine, every casting was documented with its shot profile, to monitor the final quality and repeatability. Overall 235 castings were produced with a cycle time of ~115 seconds.

In order to detect the presence of macrodefects, radiographic inspections were carried out throughout the castings and the results have been published previously [16]. This preliminary analysis evidenced how the amount, size and distribution of defects changed by changing the process parameters. Uniaxial tensile specimens were machined from the wide web, inlet and outlet walls, and ribs of the cast U-profiles as shown in Figure 2, such that the ratio length/width remained the same [16]. Some specimens were machined as aligned with the principal flow direction of the metal during die filling, while others were drawn 90° to the flow direction, as shown in Figure 2. Five sizes of flat tensile specimens were subsequently tensile tested on an MTS 810 tensile testing machine. The crosshead speed used was 2 mm/min (ε ~ 2 x 10^-3 s^-1). The strain was measured using a 25-mm extensometer. Experimental data were collected and processed to provide yield stress (YS, actually 0.2% proof stress), ultimate tensile strength (UTS) and elongation to fracture (σf). A total of 160 specimens were tested under quasi-static loading conditions at room temperature. The values of the ultimate tensile strength and elongation to fracture were converted to true tensile strength (σf') and true plastic strain (εh') through the following relationships [31]

\[
σ_f' = \frac{UTS(1+σ_f)}{1+σ_f} \tag{8}
\]

\[
σ_h' = \ln(1+σ_f) \tag{9}
\]

Finally the Quality Index, Q, was calculated as [36]

\[
Q = UTS + 0.4 \cdot K \cdot \log (100 \cdot σ_f) \tag{10}
\]

where K is the alloy’s strength coefficient.

The analysis of the fractured surfaces was carried out using an optical stereomicroscope and a scanning electron microscope (SEM). The acquired images were then transferred into a single photograph and the area of defects was quantitatively analyzed using an image analyzer (Leica LAS). The total measured defect area was then divided by the initial cross section of the tensile specimen to find the defect area fraction. In order to study the influence of casting defects on mechanical properties, the previously described constitutive and stochastic approaches were then applied and discussed.

### Table 2. Process parameters used for producing U-shaped castings

<table>
<thead>
<tr>
<th>Process</th>
<th>Plunger velocity slow shot (ms^-1)</th>
<th>Plunger velocity fast shot (ms^-1)</th>
<th>Switch point (mm)</th>
<th>Intensification pressure (bar)</th>
<th>Melt temperature in crucible (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P1</td>
<td>0.40</td>
<td>3</td>
<td>428</td>
<td>400</td>
<td>690</td>
</tr>
<tr>
<td>P2</td>
<td>0.59</td>
<td>3</td>
<td>447</td>
<td>400</td>
<td>690</td>
</tr>
<tr>
<td>P3</td>
<td>0.73</td>
<td>2</td>
<td>373</td>
<td>400</td>
<td>690</td>
</tr>
<tr>
<td>P4</td>
<td>0.58</td>
<td>2</td>
<td>451</td>
<td>400</td>
<td>690</td>
</tr>
<tr>
<td>T2</td>
<td>0.40</td>
<td>3</td>
<td>428</td>
<td>400</td>
<td>640</td>
</tr>
</tbody>
</table>
RESULTS AND DISCUSSION

ANALYSIS OF FRACTURE SURFACES

It was found that the U-shaped castings contain defects, primarily pores and oxides, and that the presence and distribution of these defects are highly sensitive to the process conditions. Significant variations of defect distribution were, however, found in castings produced under the same conditions, indicating the stochastic nature of defects in die castings [16]. The number of defects revealed on fracture surfaces varied between 2 and 20. The presence of porosity is mainly ascribed to gas entrapment phenomena during the die filling and to the blockage of vents due to the premature solidification of the molten metal. Generally, the gas pores showed a deformed spherical shape with shiny oxidized surface, while oxides appeared as rough and dull regions on the fracture surface. The presence of cold shots was generally observed, generally, incorporated within porosity (Figure 3). Contrary, sound specimens revealed a fracture mode predominantly intergranular with regions of cleavage facets, which are visible in the predominantly intergranular with regions of cleavage facets, which are visible in the interdendritic eutectic zone.

The presence of cold shots was also observed, generally, incorporated within porosity (Figure 3). Contrary, sound specimens revealed a fracture mode predominantly intergranular with regions of cleavage facets, which are visible in the silicon precipitates and brittle intermetallic phases, and with zones of deformed and fractured micronests of α-Al solid solution (Figure 4). The fracture path follows mainly the interdendritic eutectic zone.

As previously observed by Cáceres and Selling [22], the fracture surfaces were flat, although a certain degree of tortuosity was present. Therefore, trying to determine exactly if a particular defect was intersected by the fracture plane or the locus of the actual intersection was a rather arbitrary exercise. The determination of area fraction covered by defects was particularly critical for oxides, which were sometimes sitting along the main axis of tensile specimens, as shown in Figure 5. Thus, it was assumed that all visible defects on the fracture surface lied on a single cross sectional plane and the projected area was considered for the calculation.

APPLICATION OF THE CONSTITUTIVE MODEL

From tensile testing, it was observed how, considering the same investigated location of the casting, the mechanical properties, such as UTS and elongation to fracture, showed different values by changing process parameters; while, fixing the process variables, the mechanical properties changed throughout the casting. Details of mechanical properties and quality maps of diecast components are provided elsewhere [16].

In general, it was possible to observe how the different amount of defects revealed on the fracture surface influenced the tensile properties, such as UTS and elongation to fracture. As reported in Ref. 16, 21, 22 and 28, defects considerably influence the plastic tensile properties of the material but not the elastic ones. Thus, the Young modulus (E) and the YS were steady at ~72 GPa and ~177 MPa, respectively. The initial yield stress is largely determined by the relatively high supersaturation of atoms (Mg, Cu and Si) in α-Al matrix, which is referred to the high solidification rate.

The n and K values were determined by using a double logarithmic plot of the true stress and the true plastic strain, where the n-value represents the slope and the K-value corresponds to the true stress at a true strain value of unity. No relation between the defect content and the strength coefficient seems to exist, suggesting how the strength coefficient is a matrix controlled parameter. Cáceres [36] reported how the strength coefficient relates to the YS as

$$k = YS \left( \frac{E}{\alpha \cdot YS} \right)^n$$

where E is the Young modulus and α a scale factor.

Evaluating the studied alloy in terms of strain hardening exponent, it is observed that the amount of defects does not influence the plastic deformation rate, which is mainly controlled by the microstructure scale, such as secondary dendrite arms spacing [37]. The calculated n and K values were steady at 0.23 and 755 MPa, respectively.

As shown in Figure 6, the relations between the tensile properties and fractographic defects exhibit good agreement with the overall regression of experimental results. The true tensile strength (σf*) of the alloy exhibits a linear dependence that decreases from 310 to 160 MPa as the area fraction of defects increases up to about 23%. Further, the true plastic strain (εf*) decreases drastically from ~2.1 to 0.3% on an inverse parabolic relationship with the increase in defect level.
In order to model the influence of casting defects on the tensile properties, the area fraction of defects was used in the constitutive model to find the maximum homogeneous strain, ε_h^*, for the different f-values in eq. (3). Contrary to the Cáceres’ work [21], the values of true tensile strength were not normalised as defined by eq. (4), but the effect on the tensile strength was calculated using eq. (2), with n = 0.23, K = 755 MPa and the ε_h^* values from eq. (3). As shown in Figure 7, the calculated curves correctly indicate the trends of the experimental results.

Figure 7 evidences how the relation between the Quality Index and the area fraction of defects shows a good agreement with the overall regression result. The Quality Index exhibits a linear dependence that decreases from 370 to 35 MPa by increasing the f-value.

In order to model the influence of casting defects on the Quality Index, Q, eqs. (8) and (9) have been substituted into eq. (10), leading to:

\[ Q = \left( \frac{\varepsilon_h^*}{\varepsilon_{h-i}} \right) + 0.8 \cdot K \cdot \log [\exp(\varepsilon_h^*)]-1 \]  

which relates the Quality Index, Q, to the maximum homogeneous strain, ε_h^*, for the different f-values. The calculated Q-values (joined by the solid line) as a function of area fraction of defects are compared with the experimental results in Figure 7. As shown in Figure 7, the calculated curve correctly indicates, yet again, the trend of the experimental data. The results shown in Figures 6 and 7 indicate how the area fraction of defects evaluated in the fracture surface is a reliable parameter to predict the tensile properties of the material, despite some scatter in the data. The results are also in good agreement with the analytical approach based on the Ghosh constitutive model. The scatter observed in the experimental data can be considered, at least in part, as inherent to the fracture behaviour of the material [22]. Some imperfections are always present, even in sound specimens, which tend to show some scatter in tensile strength and ductility [38]. The source of horizontal scatter arises from the measurements of the defect size since the boundaries of defects were often ill-defined, making the quantification of the area of defects an arbitrary exercise. A related and more significant effect can be attributed to the fact that defects do not always lie on the same cross sectional plane, especially oxides, and therefore the projected area used to calculate f may be overestimated with respect to the real defect area fraction.

Several works [13,39,40] suggested that the morphology and the distribution of defects are important factors controlling the final mechanical properties. Thus, there is the possibility that some of the scatter can be attributed to these causes. In the present work, tensile specimens with an area fraction of defects higher than ~0.23 presented a brittle fracture well below the yield point. This means that the strain concentration in the defective region produces a very high damage rate of brittle particles within the microstructure, causing the premature fracture of the specimen. In sound Al-Si specimens, the tensile fracture generally occurs at a constant level of damage in the form of cracked eutectic Si particles, about 15% of the total particles’ population [41].

**APPLICATION OF WEIBULL STATISTICS**

The frequency distributions of UTS and $s_t$ for the five process parameters used to produce the U-shaped castings were plotted and analysed. The tensile strength and elongation to fracture data were analyzed using both the Gauss and the two-parameter Weibull models for which the parameters were estimated by the maximum likelihood method. The goodness of fit for each distribution was evaluated by the modified Anderson-Darling test, A^2. The test statistics for each fit showed that the fitted distribution could not be rejected. In general, the correlation coefficient, R^2, of the Weibull distributions calculated for the different processes is higher than ~0.98, while it is around 0.95 considering the Gauss distributions. This makes the use of the Weibull statistical approach as reliable.

As shown in Figure 8, which refers to the P1 process, both the distributions of UTS and $s_t$ are skewed about the mean values and...
they are better fitted by the Weibull distribution function than by Gauss one.

Figure 9 shows the corresponding Weibull plots where the diameter of the bubbles is proportional to the area fraction of defects evaluated in the fracture surface of the tensile specimens. While lower values in the two plots represent the specimens with the highest values, the diameter of the bubbles decreases along the straight lines. Therefore, the probability of failure $P_i$ increases by increasing the defect content $f$, indicating the fundamental role of defects on fracture mechanisms.

Table 3 shows the quantitative results of the Weibull analysis of the AlSi9Cu3(Fe) alloy cast with different process parameters. It is evident how the $\beta$ and $\eta$ values for the two-parameter fits are strongly affected by the casting parameters used. The results show that the Weibull moduli for the UTS and elongation to fracture of the P1 reference process were 9.2 and 3.3, respectively, but changing the injection profiles, as done in the P2, P3 and P4 processes, caused the Weibull moduli to decrease. The reduction of the casting temperature from 690 to 640°C in the P1 process caused a reduction of the Weibull moduli for UTS and elongation to fracture to 6.5 and 2.5, respectively. The minimum $\beta$ values were reached by using the P4 process. This means that reducing the plunger velocity during the fast shot and simultaneously delaying the commutation point between first and second phase clearly decreases the reliability of the castings.

Concerning the scale parameter $\eta$ for UTS and elongation to fracture, the highest values were reached by the P2 process, that is 256 MPa and 1.35%, respectively, while the lowest values were obtained with the P3 process where 63.21% of the specimens has failed at 230 MPa and 1.05%. These values and the highest mechanical properties reached in the work are well below the maximum mechanical properties attainable for AlSi9Cu3(Fe) alloys. Recently, Gunasegaram et al. [42] and Timelli et al. [43] evidenced how EN AC-46000 type alloys can reach values of 320 MPa for UTS and ~4% as elongation to fracture, if the defect content is significantly reduced.

In AlSi5Cu3Fe1Mg0.3 alloys modified with Mn or Sr, Zahedi et al. [30] observed that in a two-parameter Weibull plot there exists a threshold stress $\lambda$ below which failure would not occur, suggesting that three-parameter Weibull analysis would be more appropriate. When $\lambda$ is taken as zero, as in the two-parameter fits, there is a probability that the fracture stress of specimens will be less than the yield stress, even if the specimen has reached and deformed plastically beyond the yield stress. It is important to underline that in the two-parameter Weibull model, the ratio of the average to the standard deviation is a function only of $\beta$ as follows

$$\frac{\bar{\sigma}}{S_o} = \frac{\Gamma\left(1+\frac{1}{\beta}\right)}{\sqrt{\Gamma\left(1+\frac{2}{\beta}\right) - \Gamma\left(1+\frac{1}{\beta}\right)^2}}$$

where $\bar{\sigma}$ is the average fracture stress, $S_o$ is the standard deviation, and $\Gamma$ represents the gamma function. Increases in the average or decreases in the standard deviation increase the value of the Weibull modulus. Hence, higher $\beta$ does not necessarily mean higher repeatability or reliability [30].

In this work, for both UTS and elongation to fracture, however, the two-parameter Weibull was more appropriate as the three-parameter fits yielded negative threshold values or around zero. Therefore, increases in $\beta$ observed previously by changing HPDC process parameters and using two-parameter Weibull statistics could be probably considered as “an increase in safety” more than “an increase in reliability”, because it would indicate an increase in the threshold stress or elongation to fracture if three-parameter Weibull statistics were used.
AN INTEGRATED STOCHASTIC-ANALYTICAL APPROACH

The analytical approach based on the Ghosh constitutive model and the stochastic approach based on the Weibull statistics were integrated for the true tensile strength and true elongation to fracture. The tensile properties, fractographic defects and cumulative failure probability $P_i$ were correlated in Figure 8 by using 2D contour plots, which were plotted by using the Systat® v.11 software. The software first computes its own square grid of interpolated or directly estimated values. From this grid, contours are followed using the Lodwick-Whittle method combined with linear interpolation. This method is guaranteed to find proper contours if the grid is fine enough [44]. Thus, maps of iso-probability failure are generated for the tensile properties of AlSi9Cu3(Fe) alloy. Furthermore, the calculated curves using the constitutive model of tension instability were plotted as solid lines in Figure 8 to fit the experimental results, where each point is parametric to $P_i$ value. Now, it is possible to observe how 63.21% of the overall tensile specimens has failed at scale values of ~245 MPa and ~1.2%, for $\sigma_f^*$ and $\epsilon_i^*$ respectively. By intercepting the calculated solid curves, these values correspond to a defect area fraction $f$ equal to ~0.06. From another point of view, this means that tensile specimens die cast with AlSi9Cu3(Fe) alloy and with an area fraction of defects lower than ~6% have a survival probability of 36.79%.

CONCLUSIONS

The influence of casting defects on mechanical properties of a high pressure die cast AlSi9Cu3(Fe) has been investigated through a systematic experimental approach, with a theoretical verification based on constitutive and stochastic models. Based on the results obtained in the study, the following conclusions can be drawn.

- U-shaped components diecast with a combination of different injection parameters and pouring temperatures show defects mainly in the form of pores and oxides.
- The presence and distribution of these defects are highly sensitive to the process conditions. Significant variations of the defect amount and distribution are, however, found in castings produced under the same conditions, indicating the stochastic nature of defects.
- The different type and amount of defects influence considerably the plastic tensile properties of the material, such as the ultimate tensile strength and elongation to fracture, but not the elastic characteristics.
- The relations between the tensile properties and the area fraction of defects revealed on the fracture surfaces exhibit good agreement with the overall regression of experimental results.
- The analytical approach based on the constitutive model of tension instability correctly indicate the trends of the experimental results.
- The Weibull statistics evidenced how the scale parameter $\eta$ and the Weibull modulus $\beta$ are strongly affected by the diecasting parameters used, and therefore by the overall amount of defects.
- An integrated stochastic-analytical approach appears to be used to describe the tensile properties in terms of fractographic defects and cumulative failure probability $P_i$.

ACKNOWLEDGEMENTS

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Table 3. Weibull moduli, $\beta$, and scale parameters, $\eta$, for UTS and elongation to fracture values obtained from different casting processes; coefficients of determination, $R^2$, are given.

<table>
<thead>
<tr>
<th>Process</th>
<th>$\beta$</th>
<th>$\eta$ (MPa)</th>
<th>$R^2$</th>
<th>$\beta$</th>
<th>$\eta$ (%)</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>P1</td>
<td>9.2</td>
<td>249</td>
<td>0.99</td>
<td>3.3</td>
<td>1.10</td>
<td>0.99</td>
</tr>
<tr>
<td>P2</td>
<td>7.1</td>
<td>256</td>
<td>0.99</td>
<td>2.6</td>
<td>1.35</td>
<td>0.99</td>
</tr>
<tr>
<td>P3</td>
<td>8.7</td>
<td>230</td>
<td>0.97</td>
<td>3.0</td>
<td>1.05</td>
<td>0.98</td>
</tr>
<tr>
<td>P4</td>
<td>6.3</td>
<td>243</td>
<td>0.99</td>
<td>2.2</td>
<td>1.21</td>
<td>0.98</td>
</tr>
<tr>
<td>T2</td>
<td>6.5</td>
<td>243</td>
<td>0.99</td>
<td>2.5</td>
<td>1.18</td>
<td>0.99</td>
</tr>
</tbody>
</table>

Fig. 10: Contour plots of (a) true tensile strength and (b) the true elongation to fracture as a function of the area fraction of defects in the fracture surface. Each point is parametric to the cumulative failure probability $P_i$ value. The solid lines represent the calculated curves using the constitutive model of tension instability.
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Influence of the solidification microstructure and porosity on the fatigue strength of Al-Si-Mg casting alloys

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ABSTRACT
The fatigue behavior of Al-Si-Mg casting alloys is affected by the solidification microstructure and especially by defects such as gas pores, shrinkage pores and oxide films. This paper reports the microstructural characterization of a die cast engine head made of the A356 (G-AlSi7Mg0.3) alloy and the long-life fatigue strength determination using specimens extracted from the cast. The microstructural characterization was achieved by optical microscopy and digital image analysis to identify the different classes of solidification defects and to evaluate the correlation between local SDAS and the size and shape of the defects. Fatigue testing was performed under rotating bending and the stress amplitude vs cycles to failure dependence showed a large scatter. The reason for this is related to local stress concentration induced by the pores, which was analyzed by the finite element method, developing models of a material volume containing solidification pores characterized by 2D and 3D techniques (i.e. optical microscopy with image analysis and X-ray tomography, respectively).

INTRODUCTION
The A356 alloy is a classic Al-Si-Mg casting alloy that can be age hardened by solution heat-treatment, quenching and aging (T6 condition). The A356-T6 alloy is widely used for the production of engine blocks and engine heads. There is a large amount of data in literature showing that the mechanical properties of Al-Si casting alloys are largely influenced by local microstructural features, which are strictly dependent on the chemical composition and local solidification conditions [1-10]. The casting process, in particular, inevitably introduces solidification defects, which can significantly reduce the mechanical properties and, above all, the fatigue strength of the final cast component, being elements of discontinuity and acting as stress concentrators. In the case of A356/A357 alloys, previous studies [5,6,11-14] have showed that among the solidification defects, gas porosity (typically spherical in shape), shrinkage cavities (of irregular and branched shape) and oxide films (bifilm) have a dominant effect on the fatigue behavior. The influence of these defects on the fatigue strength of the A356-T6 alloy was studied by Wang et al. [5] with repeated tests being carried out at fixed values of stress.

KEYWORDS
Al-Si-Mg alloys, fatigue, defects, X-ray tomography, finite element analysis

Acronyms. Optical Microscopy: OM; Image Analysis: IA; X-ray computer tomography: XCT; hot isostatic pressing: HIP; secondary dendrite arm spacing: SDAS; stress amplitude vs cycles to failure: S/N; stress concentration factor: Kt; Linear Elastic Fracture Mechanics: LEFM.
amplitude. After grouping the test results with the same kind of defect as a fatigue crack initiator, the following important conclusions were obtained, [6]:

- Porosity (without distinction between gas porosity and shrinkage cavities) is the defect that mainly affects fatigue life;
- Only when the porosity is negligible (as in the case of castings subjected to hot isostatic pressing, HIP), the negative effect on the fatigue life of others solidification defects (such as oxide films) becomes appreciable. For example, the fatigue life of failed specimens with cracks nucleated at oxide films is 4-5 times longer than that of specimens failed by porosity;
- In defect-free specimens, the fatigue cracks generally nucleate at slip bands, eutectic Si particles and/or at intermetallic compounds. The resulting fatigue life is at least 25 times longer than that associated to cracks nucleated at pores.

The importance of reducing the size of defects, in particular casting pores, is clear and confirmed by all the literature data, which also show that when the size of the defect is reduced below a certain threshold, it does not correspond to an increase in fatigue life. The fatigue performance is directly related to the defect size and position: the greater the size and proximity to the free surface, the shorter the fatigue life of the component [11-15]. The concept of a failure-dominant pore (i.e. pore dominant for the initiation of cracks) that leads to fatigue failure is thus introduced.

Generally, the solidification defects mentioned earlier are almost always at least one order of magnitude larger than the microstructural constituents, thereby regulating the fatigue behavior of casting aluminum alloys. Only if their presence is negligible or their size comparable with that of the microstructural constituents, the latter may also influence the fatigue behavior of casting Al-Si alloys. The microstructural parameters that can mainly affect their fatigue behavior are: SDAS, size and shape of the eutectic Si particles and Fe-based intermetallic compounds [16-18].

This paper reports the results of microstructural characterization and fatigue testing carried out on specimens directly taken from different positions of engine heads, produced by gravity die casting with the A356 alloy. The microstructural characterization was aimed to identify the main classes of solidification defects and to assess any correlation between the size and shape of defects and the main microstructural parameters, which strongly depend on the local solidification conditions. The goal was to evaluate the negative influence of porosity on the fatigue strength of the A356 alloy, by performing an evaluation of the local stress concentration associated with this defect. This stress concentration was theoretically analyzed by the finite element method, developing models of a material volume containing solidification pores characterized by 2D and 3D techniques (i.e. OM with IA and XCT, respectively).

**EXPERIMENTAL PROCEDURE**

Microstructural analysis was carried out on A356 (G-AlSi7Mg0.3) specimens extracted from V8 engine heads, which were produced by gravity die casting. The alloy, which chemical composition is reported in Tab. 1, was refined with Ti-B, modified with Sr and degassed using a rotary lance degasser and high purity Ar. The casting was T6 heat-treated, solutioned at 535°C for 4.5 hours, water quenched and aged at 165°C for 4.5 hours.

The microstructural characterization was carried out by OM and IA in order to determine the SDAS and the following parameters related to the solidification defects: the percentage defects area fraction and some data related to the size and shape of the pore of maximum area, such as the equivalent diameter

\[ D_{eq} = \left( \frac{4 \cdot \text{Area}_{\text{defect}}}{\pi} \right)^{\frac{1}{2}} \]

and roundness \( R = \frac{[\text{Perimeter}_{\text{defect}}]^2}{4 \cdot \pi \cdot \text{Area}_{\text{defect}}} \)

and Féret diameter (maximum size of a hypothetical rectangle surrounding the defect).

High cycle fatigue tests, using a reduced stair-case with test interruption at 107 cycles, were performed in order to quantify the role of pores on material strength and the predictive fatigue model, based on the mechanics of defects. Tests were carried out at room temperature, because the cylinder heads may experience fatigue failure of high number of cycles in areas of moderate temperature (i.e. less than 130°C). The type of specimen loading was rotating bending (at 50 Hz). The fatigue specimens were extracted from various parts of the engine heads. After the tests were completed, analysis of fracture surfaces was carried out by SEM, which confirmed the key role of large pores. The point of failure initiation was identified and both the critical pore size and distance from the free surface were determined.

To support an investigation of the effect of pore morphology on local stress concentration in the investigated alloy, two alternative techniques were used: i) OM; ii) XCT. The first technique determines the pore shape on a 2D section with fine spatial detail, the second one is a novel technique capable of reconstructing the 3D shape of a pore inside a small volume of material [19]. The XCT analysis of solidification pores was carried out at Elettra Synchrotron in Trieste. The digital reconstruction of the geometry of shrinkage cavities was carried out with in-house software [20]. The reconstructed pores were used to create finite element models of a material volume containing realistic porosity and to calculate the local stress state [21].

**Table 1. Chemical composition of the A356 Al alloy (weight %)**

<table>
<thead>
<tr>
<th>Element</th>
<th>Si</th>
<th>Mg</th>
<th>Fe</th>
<th>Cu</th>
<th>Mn</th>
<th>Zn</th>
<th>Ti</th>
<th>Sr</th>
<th>B</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>7.24</td>
<td>0.42</td>
<td>0.138</td>
<td>&lt;0.001</td>
<td>0.007</td>
<td>0.003</td>
<td>0.120</td>
<td>0.015</td>
<td>0.0354</td>
<td>Bal.</td>
</tr>
</tbody>
</table>
RESULTS AND DISCUSSION
CHARACTERIZATION OF MICROSTRUCTURE AND SOLIDIFICATION DEFECTS

The metallographic characterization of samples machined from the engine head, showed the typical solidification microstructure of a hypoeutectic Al-Si-Mg alloy, with dendrites of primary \( \alpha\)-Al phase surrounded by the eutectic Al-Si. Solidification defects such as gas pores (basically round in shape) and interdendritic shrinkage cavities (irregularly shaped branches), were also detected. The OM and SEM micrographs, Fig. 1, did not show the presence of oxide film (i.e. bifilm).

The percentage area of defects and some parameters related to the shape and size of the largest defects (i.e. equivalent diameter, roundness and Féret diameter), were evaluated with image analysis techniques, as described in Section 2. In this regard, microstructural analysis by OM can lead to an underestimation of the actual size of the defect, in relation to the position of the cutting plane for metallographic analysis compared to the actual defect [22]. For example, an optical micrograph of a shrinkage cavity section is shown in Fig. 2-a and XCT three-dimensional image, of the same type of defect, is reported in Fig. 2-b. Regarding the analysis of the defects, particularly low values of their percentage area (ranging from 0.02% and 0.34%) have been observed at the flame deck of the engine head. A critical area was, however, the central part of the cast, where the percentage reached the upper limit of about 1.5%, reflecting both the presence of sand cores and reduced feeding by the risers. Considering the value distribution of defects percentage area fraction and equivalent diameter, about 90% of the defect area values fall below 0.75% (Fig. 3-a) and about 90% of defects have a maximum equivalent diameter between 50 and 250 \( \mu m \) (Fig. 3-b).

Weibull charts were used to characterize the statistical dependence of pore size measurements obtained by metallography. This analysis, which can be used for estimation of the expected pore sizes in larger volumes of materials, showed that the two data set of percentage area fraction of defects (corresponding to lower and higher SDAS) are aligned (Fig. 4-a) following roughly a single Weibull distribution. This superposition of data, in the Weibull chart, demonstrates that SDAS value does not seem to affect the percentage area fraction of defects in the castings under investigation. The equivalent diameter data (Fig. 4-b) appear instead influenced by SDAS: lower values are found where SDAS is lower. Moreover the two set of data (corresponding to lower and higher SDAS) are not aligned in the chart, but follow two different Weibull distributions for values of \( D_{eq} \) lower or higher than approximately 100 \( \mu m \), respectively. This trend can be related to a different genesis of the two classes of defects (with \( D_{eq} \) lower and higher than 100 \( \mu m \), respectively). The presence of a threshold size below which the pore is not fatigue-critical was proposed in [13] and confirmed in [23]. Metallographic investigation is technically limited to a characterization of the porosity on restricted areas. However, thanks to a reference statistical distribution, as identified in Fig. 4b, the maximum pore...
size expected in large section areas, with a direct link to fatigue strength, can be estimated [24].

It is well known that the mechanical properties of Al-Si alloy castings are also dependent on the values of SDAS. SDAS (µm) is directly related to the solidification rate (R [°C/s]), following the relationship: SDAS=k × R^m, where k and m are material constants (equal to 39.4 and -0.317, respectively, for alloy A356). In the analyzed cast component, the minimum SDAS (30 µm) was found at the flame deck, where the solidification rate is higher due to the presence of suitable cooling systems, while the maximum SDAS value (70 µm) was measured in the central part, which is close to the sprue or surrounded by sand cores. The casting process simulation codes are able to determine the values of SDAS with high accuracy [25], while the prediction of the characteristics of the solidification defects can be more problematic. Correlations between the defect characteristics and SDAS were so investigated. The results, reported in the plots of Fig. 5, show the lack of correlation between SDAS and % area fraction of defects, as well as between SDAS and Férét diameter. Fig. 6a also shows the lack of correlation between the roundness and Férét diameter of the largest pores, while a reasonable correlation between Férét diameter and % area fraction of defects is shown in Fig. 6b. Such a correlation, obtained by metallographic analysis, supports the use of simulation software for determining local defect size via % area fraction calculation. In addition, it is well known that fatigue behaviour of Al-Si cast components mainly depends on the size of the defects. For this reason, the existence of a correlation between Férét and % area fraction of defect should be useful in predicting the fatigue behavior of component.

**FATIGUE BEHAVIOR IN THE PRESENCE OF DEFECTS**

Data from rotating bending tests, on samples extracted from experimental castings and actual cast components, are presented in the S/N (stress amplitude vs cycles to failure) diagram of Fig. 7. Data points located at 10^7 cycles define multiple run-out, (i.e. specimens that reached the number of cycles without breaking). The scatter of fatigue life is the result of the presence of cast porosity in the A356 samples. The data scatter is large with min-max range of fatigue strength values at 10^7 cycles of 30 MPa - 75 MPa and an average value of fatigue strength of about 50 MPa. To quantify the role of the type of loading applied in long life fatigue test, push-pull tests were performed on broken specimens previously tested in rotating bending, [26]. The fatigue resistance in push-pull was found to be about 15% lower than in rotating bending due to the different material volumes under high stress.

SEM analyses of fracture surfaces confirmed the key role of porosity, which was always the point of fatigue crack initiation. Fig. 8 shows the pore of irregular shape, near the free surface of the sample, initiating the crack propagation. The crack propagated by taking a semi-elliptical shape up to the critical depth, which then led to the collapse.

**MORPHOLOGY OF DEFECTS AND STRESS CONCENTRATION**

The linear elastic fracture mechanics interpretation systematically includes the role of defects through the parameter K (stress intensity factor). K describes the elastic stress field at the tip of a crack and it is applicable when the crack length is considerably greater than the plastic zone size that inevitably forms at a crack tip in an elasto-plastic material. In addition to the nominal stress, K depends on the square root of crack length.

Recently, Shyam et al. [27,28] have interpreted the fatigue life initiated from a pore in a cast Al alloy as a small fatigue crack growth problem. He replaced the stress intensity factor range ∆K of the LEFM approach with a parameter that is the product of the monotonic and cyclic crack-tip displacements and the yield stress of the material.
material. He also demonstrated the applicability of the model to cast aluminum alloys over a wide range of experimental variables by conducting fatigue crack growth experiments from a micro-notch produced by pulse laser machining resembling a pore. Independently of the approach, whether long or short crack, the scatter in results of the fatigue testing of cast AlSi alloy highlights the need to define the equivalent crack length of a pore. Therefore, an equivalent pore size \( (\text{Area})^{1/2} \), measured on micrographs, was proposed in [24] as the initial crack size and widely used for fatigue life calculations using the LEFM approach.

The \( (\text{Area})^{1/2} \) parameter, however, is insensitive to pore morphology. When the pore is rounded (i.e. gas pore or non-metallic inclusions), its equivalent size is proportional to the radius. According to this definition, in case of elongated pores, such as a shrinkage cavity, the equivalent pore size, is much smaller than the Féret diameter. Moreover, the 2D micrographs often do not reveal the nature of typical tortuous and branched shrinkage cavity, as can be seen by comparing Figs. 2-a and 2-b.

The above issues have motivated studies based on the finite element method on the role of pore morphology as a response to high cycle fatigue life scatter. FEM models of microstructures and defects subjected to fatigue have been proposed [14, 29]. The analysis was carried out in the elastoplastic regime, since the phenomenon of fatigue damage is related to the development of microplastic deformation. The morphology of the porosity was defined by experimental evidence obtained by: i) OM and ii) XCT [19].

**STATE OF STRESS AT DEFECTS CHARACTERIZED BY METALLOGRAPHIC ANALYSIS**

Fig. 9 shows a typical shrinkage pore, whose area corresponds to equivalent size \( (\text{Area})^{1/2} = 88 \, \mu\text{m} \). Initially, the analysis in the elastic range allows the stress concentration factor \( K_t \) to be determined, which is the ratio between local maximum stress and nominal stress. Fig. 10 shows the stress map around the pore loaded in a direction perpendicular to its maximum size leading to \( K_t = 8.8 \). Further investigation revealed that the \( K_t \) for shrinkage pores is always greater than 5, in some cases reaching values of 10. The high values of \( K_t \) show that the stress levels typical of high cycle fatigue (i.e. 40-60 MPa in Fig 7), will always develop microplastic deformation at pores. Elastoplastic incremental analysis up to the nominal stress of 70 MPa, led to the development of the plastic zone at pore notches highlighted in Fig. 10. The same
image also shows the maximum pore size (Féret diameter) of almost twice the value of \((A)^{1/2}\). The morphology of the pore is very similar to a crack considered by fracture mechanics. Furthermore, the extension of the plastic zone was determined to depend non-linearly on the nominal stress, similar to models of elastoplastic fracture mechanics (i.e. for Cottrell Bilby Swinden (BCS)), [23].

**STATE OF STRESS AT DEFECTS CHARACTERIZED BY X-RAY TOMOGRAPHY**

A 3D reconstruction algorithm was developed to perform the tomography analysis of solidification defects, whose results were used to generate a finite element model of a volume of material with a microshrinkage cavity [21]. Fig. 11 shows the complex shape of the shrinkage pore reconstructed by XCT and the corresponding state of stress due to the application of a nominal stress of 70 MPa. The irregularities of the pore surface lead to strong fluctuations and local stress concentrations, as shown in Fig. 11. Maximum stress always develops in areas of minimum radius of curvature, calculated on any plane containing the direction of loading. It is reasonable to assume that this minimum curvature of the outer surface of a pore is related to the effect of surface tension of liquid alloy during the solidification process. Therefore, the formation of sharp edges is not possible. The average value of \(K_t\) for different pore orientations to the direction of load, was found to be equal to 3.2, with a variation of 15% in dependence of the orientation direction of loading. The \(K_t\) for a typical pore modeled in 3D is therefore lower than the values reported previously by 2D analysis. Figure 12 shows the accumulated plastic strain at nominally elastic stress of 70 MPa. A markedly three-dimensional effect is demonstrated with severe concentrations at points of maximum curvature of the pore surface.

**CONCLUSIONS**

This study confirmed the negative influence of porosity (cavity shrinkage and gas pores) on the fatigue strength of Al-Si casting alloys on the basis of: i) a comprehensive characterization of microstructural features and solidification defect on a cross-section of an A356 permanent mould cast engine head, ii) extensive fatigue testing on samples extracted from castings and iii) the stress analysis of casting defects. The following conclusions were reached:

- solidification conditions of the engine head are very different from area to area, leading to a variable microstructure, as shown by the local values of SDAS;
- porosity is not directly related to the local values of SDAS;
- large shrinkage pores promote the initiation of fatigue cracks, leading to premature failure of the samples with a high dispersion of data due to variability in the critical pore size;
- the damaging effect of a pore is related to local stress concentration, which was...
determined computationally for realistic shrinkage pores characterized with 2D (OM) and 3D (XCT) techniques.

ACKNOWLEDGEMENTS

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INTRODUCTION

Dislocation aggregates are examined as in hot working with some clarification from observations in creep and cold working that have been examined earlier and in greater frequency. The techniques include X-ray diffraction (XRD), etched optical microscopy (EOM), polarized optical microscopy (POM), transmission electron microscopy (TEM) scanning STEM, scanning electron microscopy - electron back scattering (SEM-EBS) and orientation imaging microscopy (OIM), with which the author has experience. Of these, only TEM images dislocations directly; the others depend on misorientations $\psi$ on opposite sides of dislocation walls. Strain induced boundaries (SIB) consisting of dislocations, serve as barriers, trapping or releasing dislocations; they may build up by accretion and merging to create high-angle boundaries that may develop high mobility in nucleation. The following mechanisms during straining at elevated temperature $T$

ABSTRACT

Substructure characteristics in hot worked Al alloys are very important for modeling mechanical properties during hot forming, and also in the product. In contrast to simple grain shape in etched-optical microscopy (EOM), polarized optical microscopy (POM) significantly confirmed subgrain presence in better detail than x-ray diffraction (XRD). Transmission electron microscopy (TEM) revealed the dislocations forming subgrain boundaries (SGB) and dispersed between them; TEM in scanning mode (STEM) could provide microtextures substantiating XRD. Scanning electron microscopy with back-scattered image (SEM-EBSI) exhibited substructures more accurately than POM but much less detailed than TEM. Finally, orientation-imaging microscopy (OIM) provided microstructures as in SEM-EBSI and also detailed misorientations; however, omission of very-low angle SGB seen in TEM gave rise to estimates of larger subgrain sizes and misorientations. The field of view is very limited in TEM, but fairly similar in POM, SEM-EBSI and OIM although higher magnifications are possible in the last two. The various techniques are also affected differently by substructure scale (temperature, strain and rate) and composition that also influence specimen preparation. Examination by several techniques is best assurance of correct interpretation of microstructural characteristics.

RIASSUNTO

Nel corso della lavorazione a caldo delle leghe di alluminio, la conoscenza delle caratteristiche della sottostruttura si rivela molto importante per la modellazione delle proprietà meccaniche sia durante la formatura che nel prodotto finale. In contrasto con la semplice forma del grano, rilevata con microscopia ottica attaccate chimicamente (EOM), la microscopia ottica polarizzata (POM) evidenzia chiaramente la presenza di sottograni con maggior dettagli rispetto a quelli forniti dalla diffrazione a raggi X (XRD). La microscopia elettronica in trasmissione (TEM) mette in evidenza le dislocazioni sia organizzate in confini che disperse all’interno di sottograni (SGB); il TEM, nella versione scanner (STEM), evidenzia le micro tessiture, confermando XRD. Il microscopio elettronico a scansione, equipaggiato con rivelatori di immagini retrodiffuse (SEM-EBSI), mostra sottostrutture in modo più accurato di POM ma con meno dettagli di TEM. La microscopia ad orientazione di immagini (OIM) è in grado di evidenziare microstrutture e disorientazioni accurate come SEM-EBSI; la mancata rilevazione dei SGB caratterizzati da disorientazione molto piccola, rilevata con il TEM, ha portato in passato a stime maggiori sia delle dimensioni che della disorientazione dei grani. Il campo osservabile, piuttosto limitato nel TEM, è abbastanza simile nel POM, SEM-EBSI e OIM anche se le due ultime consentono di ottenere ingrandimenti maggiori. Le diverse tecniche sono, in modo diverso, influenzate anche dalla dimensione della sottostruttura, (funzione della temperatura, deformazione, velocità di deformazione), e composizione che anche influenza la preparazione dei provini da osservare. Le indagini effettuate con le diverse tecniche sono la miglior garanzia della corretta interpretazione delle caratteristiche microstrutturali.

KEYWORDS

were explained in Part I: dynamic recovery (DRV); dynamic recrystallization (DRX); discontinuous (dDRX); continuous (cDRX); and grain defining (gDRV) (formerly geometric gDRX) as well as those occurring afterwards, static recovery (SRV) and recrystallization (SRX).

For these theories, metallographic problems created confusions for periods of time but new techniques with different capabilities inspired reexamination leading to successful clarification.

The objectives are to look at the unit dislocation mechanisms (cross slip, climb, subgrain boundary (SGB) formation and rearrangement) that are significant in hot working dependent on temperature T and strain rate \( \dot{\varepsilon} \) that control flow stress \( \sigma \) and ductility \( \varepsilon_p \), thus defining the process practice and the substructure, grain shape/size and texture that cause product properties.

In Part I, these have been itemized in order of progression with strain and with complexity of interaction; they will be referred to by the subsection notation system found there: (DRV); (DBTB), deformation bands + transition boundaries (TB); (SRV), serrations + DRV; (DRX). The detailed sub-goals are as follows:

1. to explain the dislocation behaviors discovered by each microscopic technique in a historic context;
2. to point out the deficiencies of the techniques and explain how hypotheses were clarified by new techniques (sometimes inadequate understanding and over-enthusiasm for a novel method created inconsistencies);
3. to bring the results of the various techniques into an integrated theory of dislocation substructure formation explaining elevated T characteristics notably steady-state stress \( \sigma_s \), thus providing a guide to extended research and improved application.

**ETCHED OPTICAL MICROSCOPY (EOM) - X-RAY DIFFRACTION (XRD)**

By about 1955, EOM had provided significant information on grain shape change in deformation and on nucleation and growth of new grains in static recrystallization (SRX) during annealing. In cold worked Al and \( \alpha \)-Fe, initial softening occurred, more at low T, without any change in grain structure. XRD showed line broadening in cold work, sharpening from SRV in annealing and break up into spots in SRX [1-3]. The questions and incomplete theories of the period (such as Mehl [3], Schmid & Boas [4], Perryman [5], Barrett [2], Sachs & Van Horne [6]) have been thoroughly reviewed for their pertinence to theories of the period (such as Mehl [3], Sachs & Van Horne [6]), having been confirmed [46]. In hot worked Al, contrasting bands appeared in elongated grains, as previously noted in in extrusion and rolling. At very low strains in Al (400°C), contrasting bands appeared in some isolated grains, but being only one subgrain thick, did not remain stable or appear for \( \varepsilon > 0.1 \) [47-50]. The size was larger as T increased or \( \dot{\varepsilon} \) decreased in consistency with XRD results in creep [7, 11, 17, 20, 49, 50]. Above 350°C, the subgrains were equiaxed in specimens compressed [22], torsioned [17, 48, 51-55], rolled [14, 15] or extruded [49, 50, 56] with \( \varepsilon \) ranging from 0.7 to 10 (Part I: 1-3, DBTB). Replicas of anodized specimens examined in TEM exhibited cubes related to the lattice in SRX grains and in elongated grains extruded above 400°C, but a sinuous roped structure below 300°C.
[56]. At lower T, POM exhibits a wood grain structure at about 100X but the subgrains are too vague to be resolvable at higher magnifications [49,57]. In hot rolling that finished at 0.55 or 0.65 Tm to 90% reduction (ε = 2.3) in one pass, Al exhibited elongated grains with subgrains that were stable in annealing at T0 for many hours [14,15]. On the other hand Cu and Ni with low stacking fault energy (SFE) retained elongated grains with strain markings (EOM) only if quenched and highly refined SRX grains within seconds [15]. In torsion to high strains, Cu and Ni exhibited DRX grains on quenching (EOM) and in Al, the POM subgrains caused such GB serrations that they were mistaken for DRX grains (discussed under serrations and SGB) [17]; although this was later rescinded [58] it gave rise to confusion for many years [18]. POM of anodized Ni has shown the presence of deformation bands, of subgrains and of serrated GB when quenched under stress to prevent SRX [47]. Since POM provided shading in some degree related to underlying crystalline orientation and subgrain sizes reasonably related to straining conditions, it was utilized to estimate misorientation Ψ between subgrains. As contrast seemed to rise with strain in steady state, it was interpreted that Ψ increased [59, 60]. This gave rise to the theory that SGB played no role in defining creep strength that still persists [25, 31, 60-62], although in cold working the various dislocation walls are believed to cause strain hardening [62-64]. Studies in depth have shown that the contrast of subgrains is strongly dependent on the angle between the polarizer’s, maximizing near extinction at 90° [46]. Results from TEM at the period [14, 15, 22, 49, 50, 55, 60] and more extensive results later (Part I: 6, 7-DRV) have not conclusively resolved the matter (discussed later under serrations on SGB). POM has played, and continues to play, a valuable role in giving a large field of view of strain homogeneity (or lack of it), of grain shape and of subgrains in Al and many alloys at strains up to 4 above 350°C [48, 52, 53]. The problems of POM for Al-5Mg are discussed under SEM-EBSI that along with TEM clarified the problem.

Although SEM-EBSI appeared about 1980 much after TEM, it is discussed here because of its similarity to POM for Al; the related technique of OIM based on SEM-EB Scanning Electron Microscopy - Back Scattered Image, diffraction is considered after TEM. In a study of hot compressed Al, (ε = 0.7, 20-500°C, 0.1-200s-1), the SEM images were fairly similar in appearance to the POM results and correlated well with the dimensions measured by TEM [65]. In a broad project, polished specimens of Al and Al-11Zn were examined first by SEM-EBSI and after anodizing by POM; regions identified by microhardness indentations coincided in detail (not the case for Al-5Mg below) [54,66-69]. A study of Al-Mg-Si, showed the development of substructure up to strains of 30; (discussed under ultraductility) [70]. In Cu that had been torsionally strained to ε = 30, EOM distinguished DRX grains (severely quenched) from SRX grains (quenched) mainly by smaller size and indistinct markings [71,72]. The SEM-EBSI and EBSD confirmed the presence of substructure in the DRX grains. SEM-EBSI of hot worked Al-5.2Mg alloy microstructures were similar to POM but with much clearer definition of each subgrain; SRX grains were clearly defined as they had been in POM of Al-1Mg earlier [51]; at 425°C 2s-1 SEM size about 7 μm compared to TEM of 3 μm [73]. In a torsion study of Al-5.2Mg at 400°C 10-3s-1, POM showed the subgrains developed at ε = 0.5 with strongly serrated GB (Part I: 9,10-DRV). The subgrains remained constant in size as the grains thinned [55]. The serrations became increasingly meandering and some appeared to pinch off, which could be mistaken for DRX [52-54,66-69]. As the grains became ever thinner, ε ≈ 10, the bollowy serrations appeared like a layer of new grains along the GB [67,69] (called rotation DRX [74]). A TEM study of all the specimens showed that the subgrains were equiaxed and constant in size and misorientation to ε = 16 at fracture [55]. The POM subgrain size was not determined so it was not realized that they were about 4x bigger than the TEM size; this was only confirmed much later [55,67-69]. In another set of experiments to ε = 5 at various T and ε, POM exposed subgrains that were larger as T rose, or ε and Z fell, in the extreme, being larger than the grain thickness at low T, high ε or Z, similar to Al even as to size [52, 53]. In the broad project on Al (previous page including Al-11Zn, Al-4.5Mg-0.7Mn), POM subgrains were similar in size in Al and Al-5Mg, but the SEM-EBSI subgrains in Al-5Mg were smaller by a factor of about 4 [54, 67-69]; the SEM ones agreed with TEM ones as explained below. TEM confirmed that the pinched off serrations contained 4-5 subgrains across a diameter; this was identical to those in the neighboring large grains [75, 76]. Clearly these were not DRX nuclei [54] confirming its absence at 5% Mg as in 1-4% as reviewed previously [77].

TRANSMISSION ELECTRON MICROSCOPY TEM

The inception of TEM in the late 1950s finally provided the ability to image individual or grouped dislocations, presenting many surprises of dislocation interactions over an ε range up to ~4. TEM was intensely applied in the 1960’s to cold working both to dislocation interactions and to the formation of substructures in an effort to explain strain hardening [28-31,63,78]. As strain increased, tangles formed and transformed into cells that decreased in size with rising strain and...
finally saturated in size (~0.5 μm) with an aspect ratio slightly above unity [79] (evidently the cell walls (CW) repeatedly rearrange, as first postulated in hot working [50]). In general, hot work researchers learned much from microstructural development both in cold working and in subsequent annealing. In the 1990's, more careful TEM exposed the formation of geometrically necessary boundaries (GNB, previously called block formation of geometrically necessary dislocations) [28, 29, 90]. The constancy of subgrain size, equiaxed shape and misorientation was confirmed in a multitude of tests up to ε = 4 [14, 15, 22, 48-50, 66-68] and in special torsion tests up to ε = 16 [55, 91], ε = 25 [92, 93], ε = 40 [94], ε = 60 [95, 96] and ε = 100 [17, 97, 98] (alternative explanations [94, 96, 99] discussed under serrations and SGB in ultra ductility). These studies, over a span of 30 years, affirmed the rearrangement of SGB by disintegration, (unknitting), reknitting and migration (Figure 2). In addition to exposing simple SGB with tilt or twist orientations, TEM has shown dislocations moving across thin foils, making deviations at SGB and undergoing cross slip in the subgrains in response to SGB stress fields [22, 49, 50]. The subgrain misorientations Ψ were confirmed as low by many neighbor-neighbor selected area diffractions (SAD) and also by rows of up to a dozen subgrains [22, 49, 50]. SAD provided Ψ across individual cellular facets for adding to micrographs; subgrains of similar orientation could be shown by dark-field illumination that was applied to Al-matrix composite exposing wide spatial scatter [100, 101] (OIM can provide even clearer information). Since GB were generally serrated (as were TB), the elongated grains (or deformation bands) could be defined only by (SAD) or by different contrasts through tilting. The presence of TB in the microstructure were determined at strains of 20, 40 and 60 by STEM Kikuchi patterns [95, 99].

The persistent equiaxed shape of the subgrains is maintained through continued rearrangement of the SGB by disintegration and reformation, migration and merging [49, 50, 86-88, 95, 96] (Figure 2). High voltage TEM of in-situ straining on a heated stage exhibited the operation of all these mechanisms [102, 103]. This behavior, named repolygonization, clearly pointed out that SGB were very transitory [50, 96, 104, 105]. Change of ε (Δε) tests clarified that the substructure rearranged into that characteristic of the new T, ε or Z conditions. The strains to steady state either initially εS (0 to εS) or in the transient (ΔεS' for ΔεS due to Δε') were proportional to change in σ [106-109]. It was proposed that in steady state repolygonization is complete each Δε = εS. In hind sight, the TEM microscopy tended to avoid GB in order to report the pure dislocation interactions that had happened inside the grains; there was similar neglect of transition boundaries (TB) between deformation bands (identifiable like grains by contrast in tilting and selected area diffraction, SAD) that being permanent have become serrated and lengthened like GB [95, 96, 104, 105]. The serrations of the GB in response to SGB attraction evidently rearrange in association with repolygonization; this proceeds in association with lengthening of the GB [110].

The subgrain sizes, similar in Al for POM and SEM-EBSI (as mentioned earlier), were slightly larger than those in TEM, because SGB with high values of δ having very low Ψ were insufficient to cause noticeable responses in oxide growth or diffraction intensity [54, 55, 66-69]. Examination of Al-5Mg through low strains showed initial development of planar dislocation arrays that gradually developed cross-links and finally subgrains at strains εSM well beyond mechanical steady state εSM [111, 112]. Apparently, the walls developed at strains up to εSM had retained stronger boundaries that controlled the growth of the anodized patches over crystal regions containing 5 subgrains across the diameter (SEM-EBSI agreed fairly well with TEM) [54, 55, 66-69, 111, 112]. While hot working of Al-5Mg to industrial strains always resulted in subgrains [20, 51-55, 66-69, 73, 74, 77], creep tests halted just after εSM had not [61, 113-115], but more extensive creep compression tests did so [67, 69, 113]. Mg does not lower the SFE but causes solute drag due to Cottrell atmospheres [69]. In the composite model, dislocations in Al-Mg move very slowly across the subgrains, whereas they fly across in Al even though there is considerable back stress from SGB [66, 69].
DYNAMIC RECRYSTALLIZATION (EOM, TEM, SEM-EBSI)

TEM also played a role in clearly defining classic discontinuous DRX as occurs in Cu. The detailed progress of classical discontinuous dDRX were described in Part I in stages 1, 2, 3, 4-DRX. In 90% reduction rolling of Cu that finished at 600°C (0.65Tm), with a 1-sec quench, the large original grains were thickly decorated along GB with new grains; TEM clearly showed the absence of substructure in these SRX grains [14]. In contrast, the initial grains exhibited fine subgrains with much less DRV than in similarly treated Al that did not exhibit any SRX [14]. The critical strain εc is higher for DRX than for SRX because the stress-driven dislocations hamper the developing nuclei that would form during SRV in annealing (Part I: 2-DRX) [116-118]. Torsion testing to 30 at higher T (lower ε, Z) and quenching produced fine grains that TEM confirmed as SRX; however, quenching under load just before stopping the motor (an excellent brake) produced very fine grains with a substructure, including also a number of random nuclei starting to grow [71, 72]. The relative sizes of DRX and SRX grains were consistent with mechanical measures of DRX and SRX in low C austenite [119]. TEM of stainless steels deformed in torsion before the peak and in steady state showed that flow stress was related to subgrain size in the same manner (Part I: g-DRX) [120, 121]. From tests on 301, 304, 316 and 317, the relation of d5 from TEM on Z (or εS) [122, 123] was similar to the relationship for Al Alloys [121]. The dependence of equilibrium grain size D5 on εS [124, 125] shows how dislocations reinserted into the new grains seriously curtail GB mobility (Part I: 4-DRV) [116-118, 126]; recent experiments confirm that a few lattice dislocations ending on a GB raises its migration activation energy to that for dislocation climb [105, 126, 127]. In Al given a TMP at 10^-2s^-1, nucleation in that substructure does not occur on reducing ε to 10^-3 or 10^-4 s^-1 (development of enlarged d5, Part I: 8-DRV) but with larger ε reduction, nucleation occurs with longer times and larger grains, indicating the influence of concurrent straining in making nucleation more difficult. [106].

SERRATION AND SGB AT ULTRA-HIGH STRAINS

The microstructural effects to be described were primarily noted in Al polycrystals with normal grains (100 - 200 μm) at high strains (20 - 130) easily attained in torsion at (400 - 550°C) (Part I: 6-SERV) [91-95]; the critical geometric condition is that the elongated grains have thinned down to 2-3 d5 [52, 53, 66-68, 95]. Because of the serrations with half amplitude of -d5 some neighboring GB come into contact pinching off the grains thus shortening them. The formation of refined grains containing a substructure as a result of grain and cell geometry was called geometric gDRX [95]; in light of the misunderstandings that have arisen, a better name would be grain-defining gDRV [104, 105, 127]. Because of the serrations, POM exhibits a field of subgrains (or crystallites) that completely masks the grains structure [52-55, 70, 95, 127-129]. TEM exposes the preponderance of SGB with regular dislocation arrays and maintenance of the steady state subgrain size (also confirmed by SEM-EBSI) [54, 66-69, 95]. The mechanical behavior and substructure dimensional parameters were the same at ε = 60 in specimens of 100 μm and 2000 μm; in the latter the grains were still 7 - 10 subgrains thick [95]. In a single crystal subjected to torsion, the substructure Ψ distribution remained stable as ε rose in steady state; however, several TB developed high Ψ, without nucleation [92, 130] in contrast to Cu and Ni which underwent DRX in single crystal torsion (Part I: 7-DRX) [131, 132]. An alternative analysis for single phase Al, including all SGB and TB but subtracting lengthened original GB, hypothesizes that the increase in fraction of HAB is evidence of continuous cDRX, in which Ψ of SGB rises with ε so that they became HAB that migrate to combine with each other [70, 94, 128, 129]. This theory does not explain why d5 and εS remain constant [70, 96, 127]. XRD of the surface shells (flattened after boring out) from both grain sizes gave the same texture that was analyzed as a deformation one [95, 99, 133] and not a DRX texture, as observed in Cu in steady state after the peak [134, 135]. Scanning TEM measures of the micro textures for both grain sizes were the same as the XRD. The scans indicated that, in each grain that had elongated, there were several TB that had rotated into layer bands like the GB.
In consistence with the geometric changes and elongation of GB and TB, many equiaxed subgrains are in contact with them (as serrations) so that 1/4 to 1/2 of their facets are high angle (without change in \( \alpha_3 \) or \( d_3 \)) [66, 67, 95, 96, 127]. There is also evidence that during high T straining, the slow GB migration associated with serrations also produces a net migration into grains with smaller subgrains (higher Taylor factor), so that they disappear; this accounts for texture evolution [Part I: 5-SERV] [99, 127, 133]. GB are also lost due to migration of triple junctions of the pancaked grains as a result of pinching off in the arms of an acute Y, thus the gDRV mechanism thickens the neighboring grains [85-87]. Similar gDRV (gDRX) phenomenon has been observed in ferritic steels; but one study with carbide decorated initial GB showed a network of MAB, possibly being TB [92, 136].

Experiments with varying Z on Al, Al-Mg-Si and Al-5Mg at rising T, or diminishing \( \varepsilon, Z \), resulted in development of gDRV (or gDRX) at much lower strains as predicted from the theory [52, 53, 67-69, 137]. However, at the highest T and lowest Z, the subgrains with diameter following the normal dependence on Z and flow stress are several times the expected grain thickness (as seen at lower T, higher \( \varepsilon, Z \)) (Part I: 6-SERV); moreover, they appear to remain completely equiaxed [52, 53, 67-69, 127]. Another set of experiments to \( \varepsilon > 20 \) also confirmed that the grain thickness stabilized near \( d_5 \) thus dependent on Z developing an equiaxed appearance [92, 93]. Finally, in friction stir welding of Al alloys, metal from in front of the advancing pin is sheared to one side in a crescent shaped zone with \( \varepsilon, Z \) declining with distance from the pin, due to constraints from the plates being welded. This crescent material is deposited behind the pin at high T, with \( \varepsilon \) declining to zero so that the SGB rapidly rearrange to a larger size causing the thinned grains to enlarge with no evidence of nucleation and growth [Part I: 7-SERV] [101]. In Al during steady state torsion at 400°C, 0.1s\(^{-1}\), upon reversal of strain (0.2-0.5-0.2-0), the grains returned to being equiaxed and the SGB and TB remained unchanged with S and as constant [138].

**ORIENTATION IMAGING MICROSCOPY OIM**

The OIM provides micrographs calculated from the variations in Kikuchi patterns (SEM-EBS Diffraction) from regions near 0.5 \( \mu \)m square. In torsion experiments, they were observed on polished discs (normal to radius near surface) that were later jetted to perforation for TEM observation; POM examination was later performed on neighboring regions [48, 138, 139]. The substructures were displayed with boundaries selected as: LAB 0.5-5°, MAB 5-15° and HAB 15-180° [105,141]. The HAB (possibly GB) and MAB (possibly TB) for \( \varepsilon = 0.2-2 \) form continuous boundaries, whereas the pattern of LAB (mainly SGB) is incomplete, although their fraction ranges from 0.9 to 0.5, as the strain in Al increases from 0.2 to 6 in the ranges 300 - 500°C 0.1 - 1s\(^{-1}\) [48, 139, 140] (Figures 1, 2). The cellular or subgrain sizes (regions classed as grains by the software) range over 3 - 8 \( \mu \)m, while in the background noise, while MAB exhibit some regions, sharp rises and matching drops (MAB) on opposite sides of deformation bands with SGB having \( \Psi = 0.5-5°(\text{av.2.2°}) \) but GB or TB above 15° with the neighboring elongated regions [92,139].

In addition to the above OIM features, the ability to run scans across a region to expose details of the boundaries is perhaps the most enlightening. The scan may provide point-to-point \( \Psi \) values so that LAB are like background noise, while MAB exhibit 5-10° and HAB 30-40°. The origin to point cumulative plots exhibit \( \Psi \) gradients across some regions, sharp rises and matching drops (MAB) on opposite sides of deformation bands [92] and large random changes at HAB [48]. The scans reinforce the color information so that one can piece together the evolution of grain deformation. In general, the similarities between POM and OIM micrographs are better than between TEM and OIM [48,138-141] (Figure 1).

From the OIM statistics giving dependence on \( \varepsilon \) up to 6 at 400°C 0.1s\(^{-1}\), the changes in fractions of LAB, MAB and HAB support the theory that \( \Psi \) of LAB saturate at about 4° [48,138-140]. From \( \varepsilon = 0.2 \) to 6, the LAB fraction decrease from 0.8 to 0.5 causing the matching HAB increment, while the MAB remain almost constant near 0.2. This has been explained in Part I: 4-SERV [98,104,105,127,140]. In reverse straining (\( \varepsilon = 0.2-0.5-0.2-0 \)), as the grains return to being equiaxed, the HAB fraction decreases, raising the LAB fraction with little change for MAB; the TB and SGB remain in existence as \( \varepsilon \) returns to 0. Both forward (as high as \( \varepsilon = 6 \)) and reverse strain take place at constant \( \alpha_3 \) and \( d_3 \) in TEM (Figure 2). The simple OIM micrographs of boundaries distinguished in groups (LAB, MAB, HAB) provide ambiguous evidence open to opposing interpretation [70]. The omission of many very low \( \Psi \) LAB causes differences between OIM and TEM; nevertheless, analysis of the changes in statistics for LAB, MAB and HAB with rising strain confirm that LAB are not marching upwards in \( \Psi \) but are being replaced by TB or GB as the number of contiguous subgrains rises with elongation. These analyses are clarified in recent reviews [104, 105, 127] and in others with broader analyses [85-87, 96, 127, 142].

**SUMMARY**

By breaking up the total phenomena of DRV behavior as it proceeds across extreme strains notably in Al and \( \sigma \) Fe alloys, it has been possible to see how the different aspects progress with strain as their distinct behaviors are driven by superimposed causes namely plastic stability (deformation bands with transition boundaries) and surface energy (serrations and pinching off). It is notable that despite significant substructural differences between high DRV hot working and cold working, the textures remain closely the same. These varied layers of mechanisms have been clarified through half a century by piecing together the results of etched and polarized light microscopy, of TEM with SAD, SEM-EBS images or diffraction and of OIM. There were apparent disagreements between the experimental techniques but
with careful analysis they can be brought into mutual clarification and confirmation of the DRV mechanism.

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