Heat treatment response and influence of overaging on mechanical properties of C355 cast aluminum alloy

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The research activity was focused on the optimization of heat treatment parameters for C355 (Al-Si-Cu-Mg) cast aluminum alloy and on its microstructural and mechanical characterization in T6 condition, also evaluating the effect of subsequent high temperature exposure. Differential thermal analyses were carried out to identify the solution heat treatment optimal temperature. After solution heat treatment and quenching, samples were subjected to artificial aging, at different times and temperatures, as to obtain the corresponding hardness curves. Samples for successive hardness and tensile tests were subjected to hot isostatic pressing (HIP) and T6 heat treatment, according to the parameters optimized in the foregoing research phase. Some of the T6 heat treated samples were also characterized after overaging, induced by holding at 210 °C for 41 h. Aiming to carry out a comparative study, tensile properties of C355 alloy, both in T6 and overaged conditions, were compared to those of A356 alloy (results from a previous study), which is currently more widely employed than C355. Experimental results showed how C355-T6 alloy is characterized by superior mechanical properties as compared to A356-T6, especially in the overaged condition, due to the higher thermal stability induced by Cu-based strengthening precipitates.

Keywords: Aluminum and alloys - Solidification - Heat treatments - Materials characterization - Metallography - Mechanical testing

INTRODUCTION

Heat treatable Al-Si-Mg cast aluminum alloys, such as A356 and A357, are widely used for the production of complex shape castings (as instance for engine applications), thanks to the low melting point and excellent castability, together with high specific strength. However, it is well known that their mechanical properties are highly dependent, not only from the casting process, but mainly on the heat treatment. It should be noted that the response to heat treatment is strongly related to each treatment phase: solution, quenching and aging, besides the waiting time between quenching and aging (i.e. pre-aging). Mechanical proper-

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Centro Interdipartimentale per la Ricerca Industriale-Meccanica Avanzata e Materiali (CIRI-MAM), Alma Mater Studiorum – Università di Bologna, V.le Risorgimento 4, 40136 Bologna ties of peak aged (T6 heat-treated) castings may be further modified by the in service conditions of the component, especially after prolonged high temperature exposure [1]. Hardness and tensile strength of A356 alloy, as instance, suffer a significant decrease at temperatures close to 200 °C, which are commonly exceeded in automotive and motorbike engine components. As to overcome this limitation, in the last years the attention has been focused on other casting alloys, such as Al-Si-Cu [2,3]. As a result of heat treatment, the presence of Cu is expected to induce the formation of strengthening precipitates with higher thermal stability, leading to the achievement of superior mechanical properties as compared to the more traditional A356/A357 (Al-Si-Mg) alloys, even after a prolonged high temperature exposure [4].

In the present work, the research activity was focused on the optimization of heat treatment parameters of C355 (Al-Si-Cu-Mg) alloy and on its microstructural and mechanical characterization, in particular aiming to evaluate the effect of high temperature exposure (overaging), by comparing its mechanical behavior with that of the A356 alloy.

MATERIALS AND METHODS

The experimental activity was carried out on C355 cast aluminum alloy, refined by the addition of a Ti-B master

alloy and modified by adding 200 ppm of Sr in the form of AISr 10 master alloy, as to spherodize and homogenize the eutectic structure. The chemical composition of the alloy, measured by a quantometer before casting operations, is reported in Table 1.

Si	Cu	Mg	Fe	Mn	Ti	В	Sr	Al
4.99	1.050	0.470	0.138	0.021	0.133	0.001	0.0212	Bal.

Tab. 1 - Chemical composition of C355 alloy (wt.%)

Tab. 1 - Composizione chimica della lega C355 (wt.%)

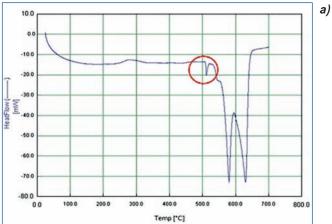
Differential scanning calorimetry analyses were carried out on as cast samples (5 mm diameter and 1,5 mm thickness) as to define the optimum conditions for the solution heat treatment. The samples were analyzed in the temperature range 20-700 °C, at 10 °C/min heating rate, in Ar atmosphere. Samples were then subjected to the solution treatment at the optimal temperature and water quenched at room temperature; then, without any pre-aging, they were artificially aged at different temperatures and times, with following hardness measurements, to determine the corresponding aging curves. Aging treatments were carried out in air furnace, with ±2°C temperature control, at temperatures ranging from 160 to 210°C for a maximum time of 12 h. For each temperature, 20 samples were aged and extracted from the furnace at predefined time intervals, then subjected to Brinell hardness measurements (performed with 2,5 mm diameter sphere and 62,5 kg load, following ASTM E 10-08 [5]). Overaging tests on peak-aged samples (T6 condition) were carried out on 6x20x4 mm samples, by holding the samples at constant temperature, in the range 170-305 °C, up to 168 h. On the overaged samples, Brinell hardness measurements were performed with the aim to obtain the corresponding overaging curves. Tensile tests were carried out in accordance to ISO 6892-1:2009 [6], on cylindrical specimens obtained by castings, subjected to HIP (Hot Isostatic Pressing) and finally T6 heat treated, according to the optimized parameters determined in the foregoing work phases. Some of the tensile specimens underwent overaging (holding at 210 °C ± 2 °C for 41 h). Both in T6 and overaged conditions, the mechanical properties of C355 alloy were compared to those of A356, characterized by similar SDAS (Secondary Dendrite Arm Spacing) values. Microstructural characterization of the studied alloy was carried out on metallographic samples, prepared according to ASTM E3-01 [7], through optical (OM) and scanning electronic microscopy equipped with energy dispersive spectroscopy (SEM/EDS). The mechanisms of failure were investigated by SEM analyses of the fracture surfaces.

RESULTS AND DISCUSSION

Heat treatment optimization

The DSC analyses on the as-cast alloy, carried out in the range 20-700 °C, highlighted the presence of an endothermic peak at about 510 °C, probably due to the melting of

the Al-Al₂Cu-Si-Al₅Cu₂Mg₈Si₄ quaternary eutectic [4, 8], as reported in Fig. 1-a. In order to avoid incipient melting, solution treatment was carried out into two subsequent steps at different temperatures; the first one, at a lower temperature, to bring in solid solution the low melting point compounds, while the second one should promote the dissolution of more stable phases. After a first solution stage (2,5 h at 490 °C), the alloy was subjected to a DSC analysis, which highlighted the absence of the endothermic peak associated to the melting of the quaternary eutectic (Fig. 1-b), confirming its complete dissolution.



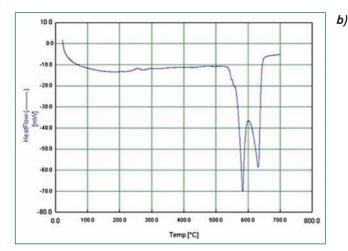


Fig. 1 - DSC curves obtained on the as cast C355 alloy (a) and after solution heat treatment at 490 °C for 2,5 h (b).

Fig. 1 - Curve DSC ottenute sulla lega as cast (a) e dopo solubilizzazione a 490 °C per 2,5 h (b).

In view of the above, the solution heat treatment was defined as follows: soaking at 490 °C for 2,5 h, subsequent temperature increase to 520 °C and soaking for 13 h, to obtain a complete solubilization. After solution treatment and water quenching at room temperature, to obtain the supersaturated solid solution, the samples were aged at different time and temperatures, as described in the foregoing section, then subjected to hardness measurements to obtain the aging curves shown in Fig. 2. The curves allowed to define the peak-aging condition (about 121 HB), reached at 180 °C for 6 h.

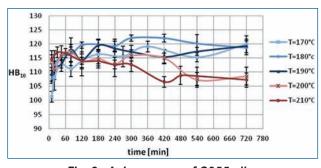


Fig. 2 - Aging curves of C355 alloy

Fig. 2 - Curve di invecchiamento della lega C355.

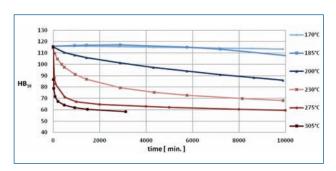
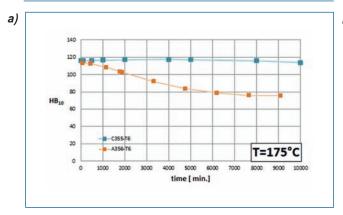


Fig. 3 - Overaging curves of C355 alloy

Fig. 3 - Curve di degrado termico della lega C355.



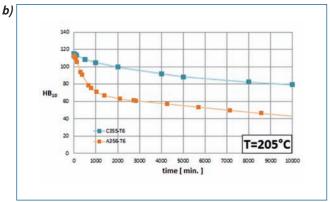
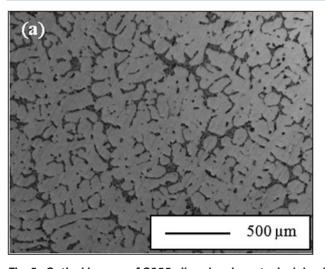


Fig. 4 - Comparison between overaging curves of C355-T6 and A356-T6 at 175 °C (a) and 205 °C (b).

Fig. 4 - Confronto tra le curve di degrado delle leghe C355-T6 e A356-T6 a 175 °C (a) e 205 °C (b).



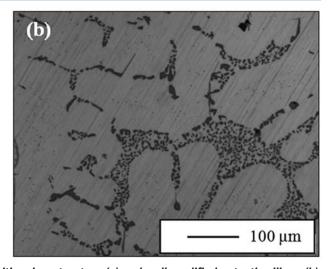


Fig. 5 - Optical images of C355 alloy showing a typical dendritic microstructure (a) and well modified eutectic silicon (b).

Fig. 5 - Micrografie ottiche della lega C355 in cui è possibile osservare la microstruttura dendritica (a) e la struttura eutettica soggetta a modifica (b).

Effect of high temperature exposure

Hardness measurements on T6 samples after overaging (induced by thermal exposure in the range 170-305 $^{\circ}$ C, for soaking time up to 168 h), enabled to obtain the corresponding overaging curves reported in Fig. 3.

Hardness of the T6 alloy was substantially unaffected by overaging up to 185°C; a slight decrease can be only observed for overaging times higher than 130 h. On the contrary, by raising temperature, the hardness decrease becomes more pronounced, reaching a minimum value of about 60 HB at 305 °C for soaking time of about 1400 min. A comparison between the overaging curves of A356-T6 and C355-T6 alloys, at two temperatures levels (175 °C and 205 °C), is shown in Fig. 4. It is possible to observe that in the peak-aged condition, the hardness is comparable between the two alloys. On the contrary, the high temperature exposure induces a much more negative

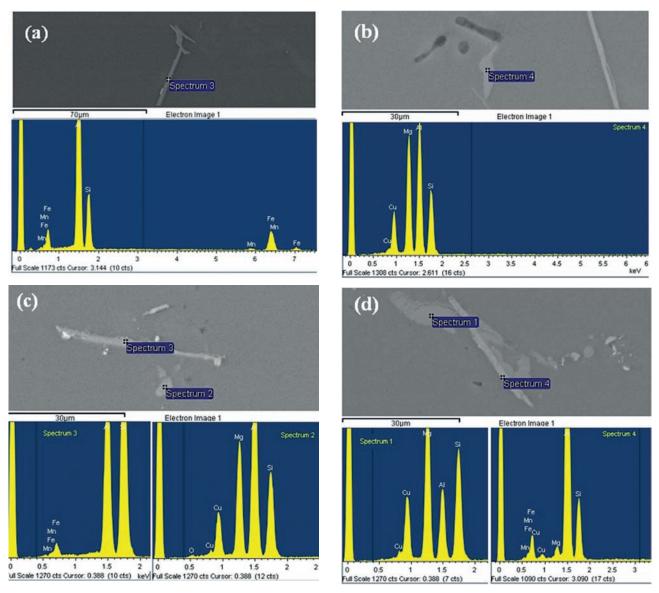


Fig. 6 - SEM images and EDS analyses on the intermetallic phases: AlMnFeSi (a) and Al₅Cu₂Mg₈Si₆ (b) on C355-T6 samples; Cu containing phases, mainly nucleated on Fe rich pre-existing phases on overaged samples (c, d).

Fig. 6 - Immagini SEM e analisi EDS su composti intermetallici: AlMnFeSi (a) e $Al_5Cu_2Mg_8Si_6$ (b) sulla lega C355-T6; composti contenenti Cu, nucleati principalmente su fasi pre-esistenti ricche in Fe, presenti su campioni sottoposti a degrado.

effect on A356-T6 alloy, as compared to C355-T6. While C355-T6 alloy keeps unchanged its hardness during exposure at 175°C, A356-T6 is subjected to a strong hardness decrease, with a reduction of about 34% (up to 76 HB) as compared to the peak value (113 HB), after 7600 min. At 205°C, it is possible to observe a progressive hardness reduction, with increasing aging time, also for C355-T6 alloy; the alloy presents a minimum hardness value of 80 HB after about 9900 min, which is however noticeably higher than the minimum hardness value exhibited by A356-T6 alloy (45 HB), at equal overaging time.

Microstructural characterization

Optical micrographs, at different magnification, of C355-T6 alloy are shown in Fig. 5. Image analyses highlighted a

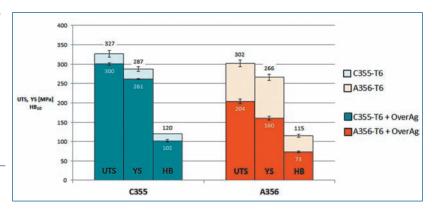
well-modified eutectic Si (class 4, according to the American Foundry Society classification system) and secondary dendrite arm spacing (SDAS) ranging from 50 to 70 μm , typical of sand castings. As expected, no appreciable differences between peak aged (T6) and overaged samples were detected.

SEM observations highlighted the presence of several intermetallic compounds, characterized by variable size (from a few to some tens of microns). Both in T6 and overaged condition, most of the large intermetallic phases found on C355 samples were identified as rod/acicular Al_5 FeSi and AlMnFeSi compounds (Fig. 6 a). The presence of these intermetallics in this alloy was already reported in the literature, both in the as-cast condition and after heat treatment [8]; the solution heat treatment temperature, in fact, is not high enough to dissolve the compounds, which

Trattamenti termici

Fig. 7 - Comparison of the mechanical properties (UTS, YS, HB) at room temperature of C355 and A356 alloys, in T6 and overaged conditions.

Fig. 7 - Confronto delle proprietà meccaniche (UTS, YS, HB) a temperatura ambiente delle leghe C355 e A356 nella condizione T6 e a seguito di degrado termico.



are the first intermetallic phases to form during solidification [8]. Even if in a lower amount as compared to Fe-rich intermetallics, Cu-containing phases, such as the ${\rm Al_5Cu_2}$ Mg₈Si₆, were also detected in C355-T6 samples (Fig. 6 b). The lower amount of detectable Cu based phases is probably related to the solution heat treatment, able to bring in solid solution Cu atoms, as to allow the subsequent precipitation of nanometric ${\rm Al_2Cu}$ compounds (not detectable by SEM) during artificial aging. The overaged samples, on

the contrary, showed a slightly more elevated quantity of Cu containing phases, detectable by SEM due to their size increase. They often nucleated on Fe-based intermetallics (Fig. 6 c, d). Even if further work is required to investigate this aspect, the presence of such large Cu-based intermetallics is likely to be related to the effect of overaging, i.e. holding at 210 °C for 41 h, which led to their coarsening through diffusion processes.

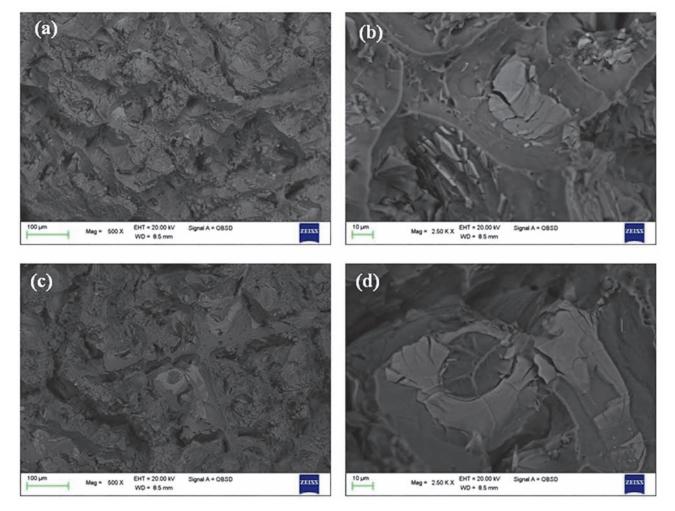


Fig. 8 - SEM images of tensile fracture surfaces of: C355-T6 alloy at low (a) and high (b) magnification; overaged C355-T6 alloy at low (c) and high (d) magnification.

Fig. 8 - Immagini SEM delle superfici di frattura: lega C355-T6 a basso (a) ed alto (b) ingrandimento; lega C355-T6 dopo degrado a basso (c) ed alto (d) ingrandimento.

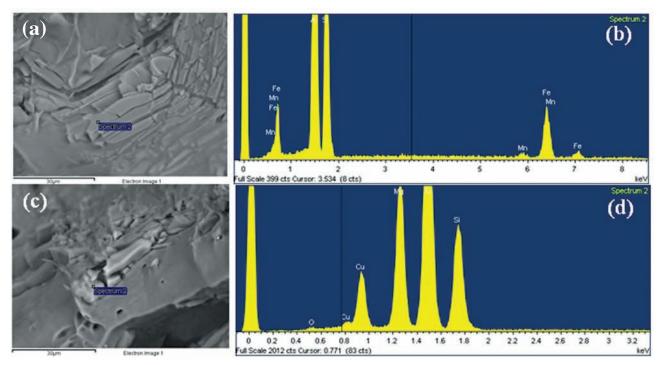


Fig. 9 - SEM images and corresponding EDS spectra of decohesed and fractured intermetallics: AlMnFeSi (a, b) and $AI_{\kappa}Cu_{\kappa}Mg_{\kappa}Si_{\kappa}$ (c, d).

Fig. 9 - Immagini SEM e corrispondenti spettri EDS di composti intermetallici: AIMnFeSi (a, b) e Al¸Cu¸Mg¸Si¸ (c, d).

Mechanical properties

The results of hardness and tensile tests on the peak aged (T6) and overaged (41 h at 210 °C) C355-T6 alloy, are reported in Fig. 7, compared with those of A356 alloy. It can be noted that the peak-aged C355-T6 alloy exhibits slightly higher mechanical properties than A356-T6; however, it is worth noting how A356-T6 alloy is much more sensitive to high temperature exposure as compared to C355-T6. As a matter of fact, after exposure at 210 °C for 41 h, while C355-T6 shows a decrease of UTS and YS of 8% and 9%, respectively, the A356-T6 shows much more relevant strength loss: UTS decreases of 32%, while YS of about 40%. Similarly, the hardness of C355-T6 decreases of about 15% as a result of overaging, while the reduction of A356-T6 is more consistent (i.e. 37%). Elongations to failure are comparable in all the tested conditions (C355-T6/A356-T6 before and after overaging), approximately equal to 5%.

Fracture surfaces analysis

Representative SEM images of the tensile fracture surfaces of C355 alloy specimens (both in T6 and overaged conditions) are shown in Fig. 8. The fracture path is prevalently transgranular; several fractured and decohesed intermetallic compounds were identified. Due to the absence of typical solidification defects, such as porosity and shrinkage cavities (eliminated by HIP treatment), the relatively low elongations to failure of the alloy were correlated to the presence of these brittle phases.

EDS analyses on the intermetallic phases found on fracture surfaces, allowed to evaluate their chemical composition (Fig. 9), confirming the results obtained by EDS

analyses on metallographic samples. Most of them were, in fact, hard and brittle Fe-based intermetallic compounds, playing a negative effect on the elongation to failure. Fracture of large intermetallics was observed, as a consequence of the matrix deformation, which induced high level of stress at their interface [9]. Fracture surfaces of overaged samples did not show noticeable differences as compared to the T6 samples. The effect of overaging on UTS, YS and, above all, on elongation to failure, is not so relevant as to justify substantial differences in the failure mechanisms between the two classes of samples.

CONCLUSIONS

In the present work, optimization of the heat treatment of C355 alloy was carried out, by defining optimal solution temperature and peak-aging parameters. The effect of long-term high temperature exposure on its hardness was also evaluated, by determining C355-T6 overaging curves. A comparison between overaging behavior of C355 and A356 alloys, by hardness and tensile tests, showed a superior thermal stability of the former, due to the presence of Cu-based strengthening phases.

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Risposta al trattamento termico e influenza del degrado sulle proprietà meccaniche della lega di alluminio C355

Parole chiave: Alluminio e leghe - Solidificazione - Tratt. termici - Caratterizz. materiali - Metallografia - Prove meccaniche

L'attività di ricerca è stata focalizzata sull'ottimizzazione dei parametri di trattamento termico della lega di alluminio da fonderia C355 (Al-Si-Cu-Mg) e sulla successiva caratterizzazione microstrutturale e meccanica in condizione T6. È stata inoltre valutata la stabilità termica della lega, a seguito di esposizione ad alte temperature, confrontandola con quella della più utilizzata lega A356 (Al-Si-Mg).

Sono state effettuate analisi termiche differenziali per identificare le temperature ottimali relative al trattamento di solubilizzazione. Al fine di evitare incipiente fusione di fasi eutettiche bassofondenti, il trattamento è stato effettuato in due fasi consequenziali: una prima fase di solubilizzazione a 490 °C per 2,5 h ed una seconda fase di solubilizzazione a 520 °C per 13 h, volta alla completa omogeneizzazione del materiale. I campioni solubilizzati e temprati sono stati successivamente sottoposti ad invecchiamento, a tempi e temperature diverse, per ricavarne le corrispondenti curve di durezza. Tali curve hanno consentito di identificare i parametri di invecchiamento al picco di durezza T6 (180 °C, 6 h).

E' stato successivamente effettuato uno studio sul degrado termico della lega, sottoponendo campioni trattati T6 a permanenze prolungate in temperatura (da 170 a 305 °C, per un tempo massimo di 168 h). Dall'analisi delle corrispondenti curve di durezza, è risultato evidente come, a confronto con la più diffusamente impiegata A356, la lega C355 presenti un decremento inferiore di durezza e dunque una maggiore stabilità termica.

I campioni per le successive caratterizzazioni meccaniche sono stati sottoposti a pressatura isostatica a caldo (HIP) e trattamento termico T6, secondo i parametri ottimizzati nella precedente fase di lavoro. Parte dei campioni trattati T6 è stata caratterizzata anche dopo degrado termico, indotto da permanenza in forno a 210 °C per 41 ore. I risultati della sperimentazione hanno evidenziato come la lega C355-T6 sia caratterizzata da proprietà meccaniche superiori rispetto alla lega A356-T6 (YS, UTS), in particolar modo in condizioni di degrado, presentando diminuzioni percentuali, rispetto alla condizione T6, di molto inferiori rispetto alla lega A356.

Le proprietà meccaniche della lega C355 sono poi state correlate alla microstruttura tramite analisi in microscopia ottica (OM) ed elettronica in scansione (SEM/EDS), unitamente all'osservazione delle superfici di frattura. Oltre alla presenza di fasi intermetalliche a base Fe, non solubilizzate durante il trattamento termico, sono stati rilevati anche precipitati intermetallici contenenti Cu, in particolar modo nei campioni sottoposti a degrado. Mentre nei campioni invecchiati al picco di durezza il rame forma precipitati di rinforzo nanometrici (dunque non osservabili al SEM), nel caso dei campioni sottoposti a degrado i fenomeni diffusivi indotti dalle alte temperature, ne hanno favorito l'accrescimento.

In conclusione, è stato possibile verificare come la presenza del rame in lega induca non solo un incremento delle proprietà meccaniche in condizione T6, ma anche una migliorata stabilità termica del materiale, caratteristica che rende la lega C355 di grande interesse per la realizzazione di getti trattabili termicamente per applicazioni ad alta temperatura.