



Effects of artificial aging conditions on mechanical properties of gravity cast B356 aluminum alloy

D. DIONI, S. CECHEL, G. CORNACCHIA, M. FACCOLI, A. PANVINI

Department of Mechanical and Industrial Engineering, University of Brescia, Via Branze 38-25123 Brescia, Italy

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Abstract: The age hardening behavior of gravity cast B356 aluminum alloy was investigated by differential scanning calorimetry (DSC), hardness measurements and tensile tests. Three different artificial aging temperatures were selected, namely 155, 165 and 180 °C, with heat treatment time from 40 min to 32 h. DSC analysis results show that cluster formation begins below room temperature (at around −10 °C). Since cluster formation influences the subsequent precipitation of the main strengthening β'' phase, it can be inferred that a delay between solutionizing and artificial aging has a detrimental effect on the mechanical properties of the alloy. It was also confirmed that the hardness and the tensile properties of the alloy reach the maximum values when β'' phase is completely developed during the artificial aging. This happens after 16 h for samples aged at 155 °C, after 6 h for samples aged at 165 °C and after 4 h for samples aged at 180 °C. A subsequent decrease of the mechanical properties, observed only in the sample aged at the highest temperature, with increasing aging time can be associated with the transformation of the coherent β'' phase into the semi-coherent β' phase. Finally, the activation energy associated with the precipitation of β'' phase was calculated to be 57.2 kJ/mol.

Key words: B356 aluminum alloy; gravity casting; artificial aging; precipitation sequence; mechanical properties

1 Introduction

Weight reduction in car production is a main topic of tomorrow generation vehicles. In order to achieve the expected results in terms of weight loss of vehicles frames, optimized structural cast aluminum parts need to be produced and assembled with wrought ones. Strength and security specifications defined by structural designers need to be fulfilled by those parts limiting as much as possible the oversizing of components to avoid overweighting. Up today, 4 mm minimum thickness for cast parts is specified by intrinsic limits of permanent mold casting technology, but the scenario is rapidly changing since new foundry processes allow very thin parts well below the 3 mm threshold. In this situation, safety margins are strongly reduced and the mechanical properties of the casting itself need to be known better and be tailored to the designer needs. Traditional T6 heat treatments need to some extent to be optimized in order to grant different balancing of strength and elongation properties according to the required performances needed by specific parts.

Al–Mg–Si alloys are a group of alloys extensively

used in the automotive industry, in both cast and wrought forms. They are precipitation hardening alloys, which are usually heat-treated to the T6 condition in order to obtain the required mechanical properties, and they undergo a very complex precipitation sequence during artificial aging. While many studies have been carried out to better understand this topic in wrought alloys, the investigation of foundry alloys has not yet received the same attention [1–12]. Although foundry and wrought alloys clearly differ in Si content, the strengthening mechanism has usually been assumed to be similar. Up today many aspects remain controversial and various theories are available in literature, especially concerning the initial cluster precipitation. EDWARDS et al [3] studied the fine-scale precipitation that occurs during age hardening of wrought Al alloy 6061, using differential scanning calorimetry (DSC), one-dimensional atom probe (1DAP) and transmission electron microscopy (TEM). DSC analysis of an as-quenched sample showed the presence of one exothermic peak up to 110 °C. The 1DAP results highlighted that three types of clusters are formed in the early stages of aging: clusters of Si atoms, clusters of Mg atoms and co-clusters that contain both Mg and Si atoms (also termed as “GP zones”). The clusters did not form

simultaneously: independent clusters of Mg and Si atoms formed at first (during or immediately after quenching), they disappeared with time, were replaced by Mg–Si co-clusters. The authors concluded that the exothermic peak observed between room temperature and 110 °C was due to co-clustering of Mg and Si atoms, and also suggested that the combination of the large exothermic peak due to Mg–Si co-clusters formation and the small endothermic peak due to the dissolution of Mg might give the appearance of a double-exothermic peak. Similar results were found by GEUSER et al [9] using the three-dimensional atom probe (3DAP) technique. Other researchers [2,10,12] observed the presence of two exothermic peaks in the temperature range of 30–100 °C. ZHEN et al [2] carried out DSC analyses of two wrought Al–Mg–Si alloys with high Si content. They suggested that these peaks can be associated with the same three types of clusters found in Ref. [3]. More recently, the combined use of DSC and 3DAP analyses was exploited by SERIZAWA et al [10] to investigate the nanoscale precipitate microstructures of a wrought Al–Mg–Si alloy. Two exothermic peaks found at approximately room temperature and 100 °C were explained by the formation of two types of Mg–Si nanoclusters, with different Mg/Si ratios. Similar results of the cast A356 aluminum alloy were found by CESCHINI et al [12]. Therefore, the current literature suggests two possible initial clustering sequence: Al SSS→independent clusters of Si and Mg atoms→co-clusters of Si and Mg atoms [2,3,9]; otherwise, Al SSS→Mg–Si nanoclusters with different Mg/Si ratios [10,12]. The cluster formation is of great interest because it directly influences the subsequent precipitation, thus a “pre-aging” at room temperature between quenching and artificial aging can influence the final mechanical properties of the alloy [3,12]. According to EDWARDS et al [3], the subsequent precipitation sequence is: co-clusters of Si and Mg atoms→small precipitates of unknown structure→ β'' phase→ $\beta'+B'$ phases→ β phase. β'' phase is considered the main strengthening phase in these alloys [3,7,12]. The last precipitation sequence is also influenced by the Si content of the alloy. The effect of excess amount of Si was studied by MURAYAMA and HONO [5] in aged wrought Al–Mg–Si alloys with various Si contents, using 3DAP and TEM. They found that the excess amount of Si forms Si precipitates after the precipitation of β'' phase.

In this work, the age hardening behavior of a foundry alloy, namely the gravity cast B356 aluminum alloy, was studied in order to optimize its mechanical properties. The influence of various artificial aging temperatures and time on the precipitation sequence was investigated by DSC. The effects of the aging condition on the mechanical properties were evaluated through

hardness and tensile tests. Afterwards, the mechanical properties of the alloy were correlated with the DSC results. Finally, the activation energy associated with the precipitation of β'' phase was calculated.

2 Experimental

The chemical composition of the studied Sr-modified B356 aluminum alloy is shown in Table 1.

Table 1 Chemical composition of B356 aluminum alloy (mass fraction, %)

Si	Fe	Cu	Mn	Mg	Ti
7.434	0.078	0.008	0.010	0.331	0.106
B	Ca	Na	Sb	Sr	Al
0.0001	0.002	0.001	0.001	0.016	Bal.

The alloy was provided as thin rectangular bars (100 mm × 24 mm × 3 mm) separately cast from automotive components by gravity casting. Specimens for DSC tests were taken from a bar prior to the heat treatment, in order to avoid alteration due to the cut [8]. They were approximately 4 mm × 4 mm × 1 mm, with an average mass of 20–25 mg. The bars and the DSC specimens were solutionized at 540 °C for 6 h in a preheated electric oven, immediately followed by water quenching at room temperature. Then, they were refrigerated at a temperature of approximately –18 °C. The time lapse between quenching and refrigeration did not exceed 5 min to avoid detrimental effects on the mechanical properties of the alloy due to natural aging [12,13]. Afterwards, they were artificially aged in a preheated oven at 155, 165 and 180 °C in a time range from 40 min to 32 h.

The influence of the aging condition on the precipitation sequence was investigated by DSC. The tests were performed using a TA Instrument DSC Q100, in a temperature range from –50 °C to 350 °C, with a heating rate of 10 °C/min and operating in nitrogen atmosphere. The thermal events associated with the phase transformation were analyzed in each sample after the subtraction from the experimental results of the baseline run performed with empty sample holders. A solution-treated sample, termed “as-quenched” to differentiate it from the aged samples, was also analyzed by DSC and its run was compared with that of the aged samples.

The effects of the aging condition on the mechanical properties of the alloy were studied through hardness and tensile tests. The specimens were machined out of the heat-treated bars. The hardness tests were carried out according to ASTM E 18–03 [14] by a hardness tester Rockwell Rupac 500Mra, with a steel indenter diameter

of 1.58 mm, a load of 588 N and a dwell time of 15 s. For each hardness value, ten measurements were taken and averaged. The samples were prepared with standard metallographic techniques (ground with SiC paper and polished with 1 μm diamond paste) and were also observed using a Leica DMI 5000M optical microscope. Tensile tests were carried out according to UNI EN ISO 6892-1(2009) [15], using an electromechanical testing machine Instron 3369 at a strain rate of 0.4 mm/min. Dumb-bell samples with a length of 48 mm, width of 12 mm and thickness of 3 mm were used.

Finally, the “as-quenched” alloy was also subjected to a DSC analysis at different heating rates in the heating rate range of 2.5–15 $^{\circ}\text{C}/\text{min}$, in order to obtain the activation energy of β'' phase precipitation according to the Kissinger method [16]. The activation energy can be considered an important parameter in the quantification of alloy age hardening behaviour [17–19] and can be determined by DSC curves. The dependence of the peak temperature of a defined transformation T_p on the heating rate α can be used to calculate the activation energy E of the process by applying the relationship:

$$\ln(\alpha/T_p^2) + E/(RT_p) = C \quad (1)$$

where R is the molar gas constant; C is the constant. By plotting $\ln(\alpha/T_p^2)$ vs $1/T_p$, the slope of the linear regression fitting will be $-E/R$ and the constant can be calculated from the intercept with the vertical axis of the linear regression line.

3 Results and discussion

3.1 DSC Analysis

Figure 1 shows the DSC curve of the “as-quenched” sample. The DSC analysis was carried out in a temperature range from -50°C to 350°C to evaluate the precipitation sequence in the B356 aluminum alloy both below and above room temperature. Few attempts were found to investigate the Al–Si–Mg alloy behavior below room temperature by thermal analysis [6,10]. Therefore,

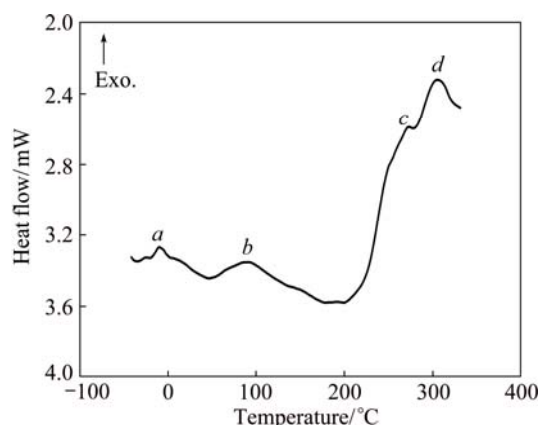


Fig. 1 DSC curve for as-quenched B356 aluminum alloy

very little data are available on the precipitation behavior of these alloys at low temperatures.

The DSC curve shows four distinct exothermic peaks: around -10°C (peak *a*), 90°C (peak *b*), 270°C (peak *c*) and 310°C (peak *d*). The peak identification of DSC thermogram of Al–Si–Mg alloys involves many techniques and is not unique in the current literatures. Therefore, it will be performed by the support of previous works [1–4,9,10,12], almost always carried out on 6xxx series alloys, focusing the most relevant information linked to the B356 alloy.

In the range from -50°C to 110°C an exothermic peak at around -10°C (peak *a*) was observed, in addition to an exothermic peak at around 90°C (peak *b*). Based on Refs. [10,12] they can be explained by the formation of two types of Mg–Si nanoclusters found by SERIZAWA et al [10]. It is worth noting that cluster formation begins below room temperature. This phenomenon has not been observed in previous works, also because the majority of the DSC analyses did not scan below room temperature [2,3,12]. Since the formation of clusters directly influences the subsequent precipitation, it is confirmed that any low temperature aging treatment or delay between quenching and artificial aging can influence the final mechanical properties of the alloy [3,12].

An exothermic peak (peak *c*) was found at around 270°C . EDWARDS et al [3] observed two superimposed exothermic peaks: one at around 215°C and the other at around 250°C . They inferred that the former is due to the formation of fine precipitates with unknown microstructure, whereas the latter is due to the precipitation of β'' phase. CESCHINI et al [12] did not observe a similar combination of two peaks in the same range of temperatures, but found only one exothermic peak at around 230°C . They explained that this effect is due to the high heating rate used in the analysis and the high Si content of the A356 Al alloy, which increases the precipitation rate. Therefore, according to Refs. [3,12] peak *c* could correspond to the transition from Mg–Si co-clusters to the main strengthening phase β'' . The absence of an endothermic peak between peaks *b* and *c* suggests that the transformation proceeds without dissolution of Mg–Si co-clusters.

An exothermic peak (peak *d*) was found at around 310°C . According to Refs. [3,12] it can be caused by the transition from β'' phase to β' and B' (Si-rich precipitate) phases. Peak *d* partially overlaps peak *c* because the β'' phase loses its coherency with the matrix and transforms to the β' phase as soon as it precipitates [7,8,11]. CESCHINI et al [12] observed a superimposition of two exothermic peaks approximately at 300°C , corresponding to the formation of β' and B' phases. No visible combination of these two peaks was detected in

the DSC curve of Fig. 1.

Figure 2 shows DSC curves for T6 heat-treated B356 alloy, with artificial aging carried out at 155 °C for various time. Curves are shifted along y-axis to help the observation.

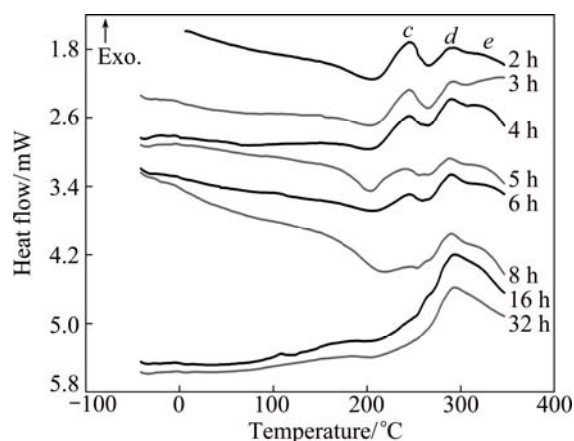


Fig. 2 DSC curves of T6 heat-treated B356 alloy aged at 155 °C for various time

The exothermic peaks *a* and *b*, observed in the DSC curve of the “as-quenched” sample, completely disappear in all DSC curves of the aged samples. This result suggests that the cluster formation occurs during the artificial aging.

The exothermic peak *c* (at around 250 °C), due to the formation of β'' phase, is observed only in some DSC curves. The height of intensity of peak *c* decreases with increasing aging time and the peak completely disappears after 16 h. This trend suggests that β'' phase is fully formed after 16 h of artificial aging. This conclusion can be drawn thanks to previous works, as that of ESMAEILI et al [20], where DSC analysis was supported by quantitative TEM. They determined the volume fraction of the precipitates in the AA6111 Al–Mg–Si–Cu alloy solutionized at 560 °C for 10 min, quenched in water at room temperature and aged at 180 °C for various time. In addition, they observed that the peak associated with the main strengthening phase β'' disappeared after aging at 180 °C for 1 h. Quantitative TEM analysis proved that the volume fraction of β'' phase increased at the beginning of the aging, reaching the maximum value after aging at 180 °C for 1 h.

The exothermic peak *d* (at around 300 °C), due to the formation of β' and β' phases, seems not to be affected by artificial aging. Furthermore, the exothermic peaks *c* and *d* in all DSC curves of aged samples are shifted towards lower temperatures compared with the DSC curve of the “as-quenched” sample. This effect is due to the artificial aging that involves an early precipitation during the DSC scans. In the DSC curves of the aged samples an additional peak *e* appears at around

330 °C for time until 16 h compared with the DSC curve of the “as-quenched” sample. According to previous works [2,4,5,11,21] this peak could be associated with Si precipitation.

The same considerations may be done for the DSC curves of samples aged at 165 °C and 180 °C for various time (Fig. 3). The only difference in the DSC curves is the aging time where the peak *c* disappears. This is estimated to be between 4 h and 8 h for the samples aged at 165 °C and between 2 h and 4 h for samples aged at 180 °C.

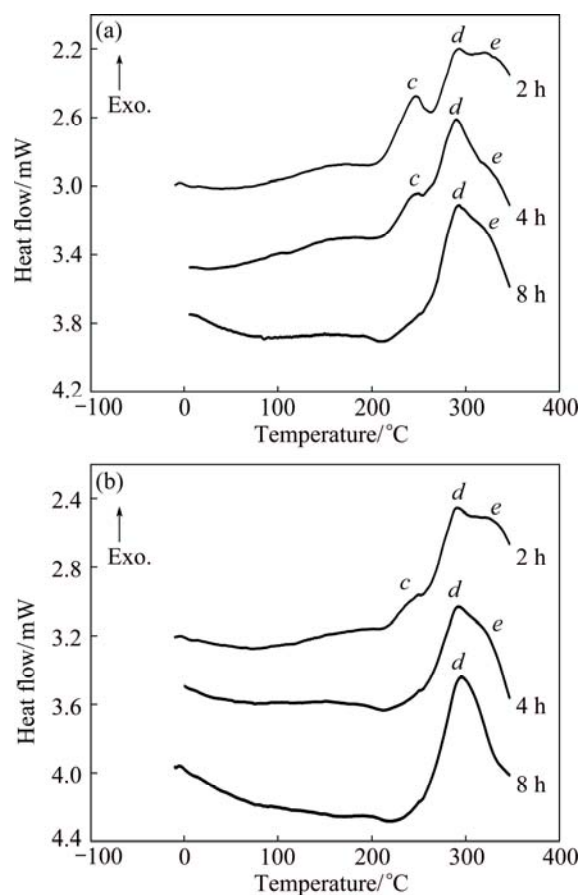


Fig. 3 DSC curves of T6 heat-treated B356 alloy aged at 165 °C (a) and 180 °C (b) for 2, 4 and 8 h, respectively

The activation energy *E* of the main strengthening phase β'' was calculated according to the Kissinger method [16] using Eq. (1) by plotting $\ln(a/T_p^2)$ versus $1/T_p$, as shown in Fig. 4.

The slope of the linear regression fitting is $-E/R$ and the activation energy is $E=57.2$ kJ/mol. The obtained *E* value is close to that found by GABER et al [22] associated with the precipitation of β'' phase (77.4 kJ/mol) in a balanced Al–Mg–Si alloy with a Si content of 0.44%. The difference between these two values could be due to the different chemical compositions of the alloys, as B356 aluminum alloy has an excess in Si content.

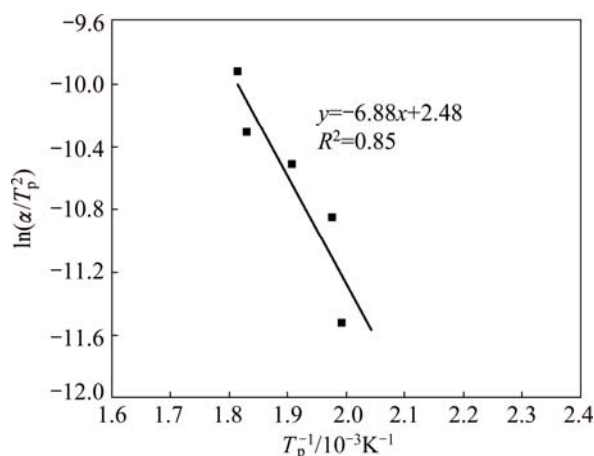


Fig. 4 $\ln(\alpha/T_p^2)$ vs T_p^{-1} for β'' phase

3.2 Mechanical properties and microstructures

The hardness of the B356 alloy as a function of aging time at various aging temperatures is shown in Fig. 5.

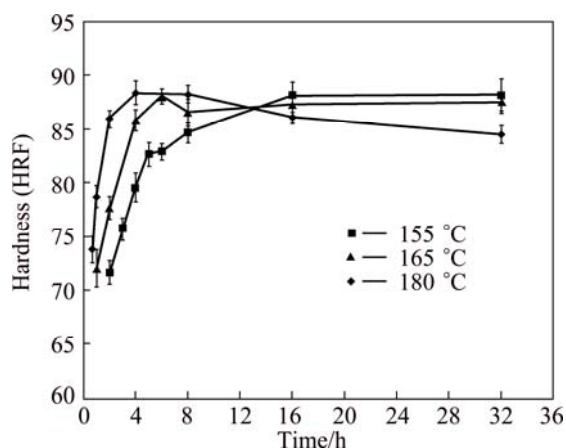


Fig. 5 Hardness of B356 alloy as function of aging time at various aging temperatures

There are two topics that is worth investigating: the effect of aging time and aging temperature on the hardness evolution. As expected, hardness increases very rapidly with increasing time at the beginning of aging for all aging temperatures. It reaches a maximum value of approximately HRF 88 at all aging temperatures and then decreases only at the highest aging temperature (180 °C). In addition, a higher aging temperature produces an earlier peak hardness compared with a lower temperature. The hardness peak at aging temperature of 180 °C reaches after 4 h, at 165 °C it reaches after 8 h and at least 16 h are required for the sample aged at 155 °C to reach the peak hardness. Higher aging temperatures increase the diffusion rate of vacancies and solute atoms, resulting in a faster kinetics of cluster formation. As a consequence, during short time the hardness of samples aged at higher temperatures is almost higher than that of

samples aged at lower temperatures. At longer aging time hardness values start to get very close to each other and finally the same maximum hardness reaches.

The results of tensile tests as a function of aging time at various aging temperatures are plotted in Fig. 6.

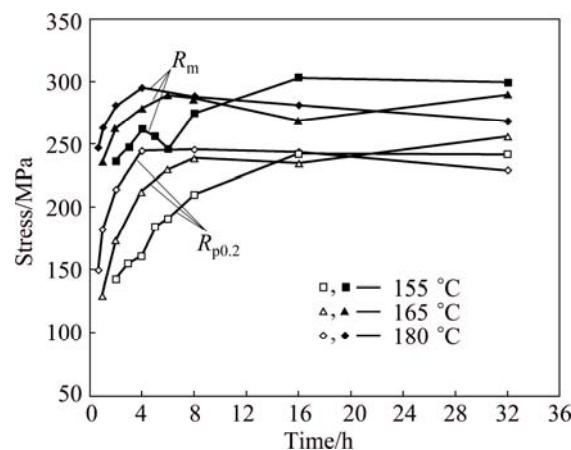


Fig. 6 Tensile properties of samples as function of aging time at various aging temperatures

By comparing Fig. 6 with Fig. 5 it can be observed that the evolution of yield strength ($R_{p0.2}$) and ultimate tensile strength (R_m) as a function of aging time is similar to that of hardness. This effect can be explained considering that the cluster formation is the mechanism responsible for changes in all these properties. A similar maximum value of $R_{p0.2}$ (~250 MPa) and a similar maximum value of R_m (~300 MPa) were found after all heat treatments at any aging temperature. A higher aging temperature leads to an earlier peak of both $R_{p0.2}$ and R_m compared with a lower aging temperature. This peak is followed by a slight decrease with increasing aging time only for the highest aging temperature (180 °C). It was assumed that the decrease in R_m of the samples aged at 155 °C for 5 and 6 h is mainly due to casting defects, such as porosity.

Finally, the microstructures of the alloy aged at 155 °C are shown in Fig. 7. It can be seen from Fig. 7 that the α (Al) dendrites and the interdendritic network of the Al–Si eutectic phase are easily identified. The Si particles of the eutectic phase show the typical globular shape due to the modification effect of Sr. No remarkable effect is observed with varying the aging time.

3.3 Relationship among results of DSC analysis, hardness and results of tensile tests

Age-hardening response and the tensile behavior of the artificially aged B356 alloy can be accounted for accurately by the DSC analysis. The reduction of peak *c* in DSC curves (Figs. 2 and 3) as well as the increase in hardness (Fig. 5) and in tensile properties (Fig. 6) with increasing aging time can be associated with the gradual

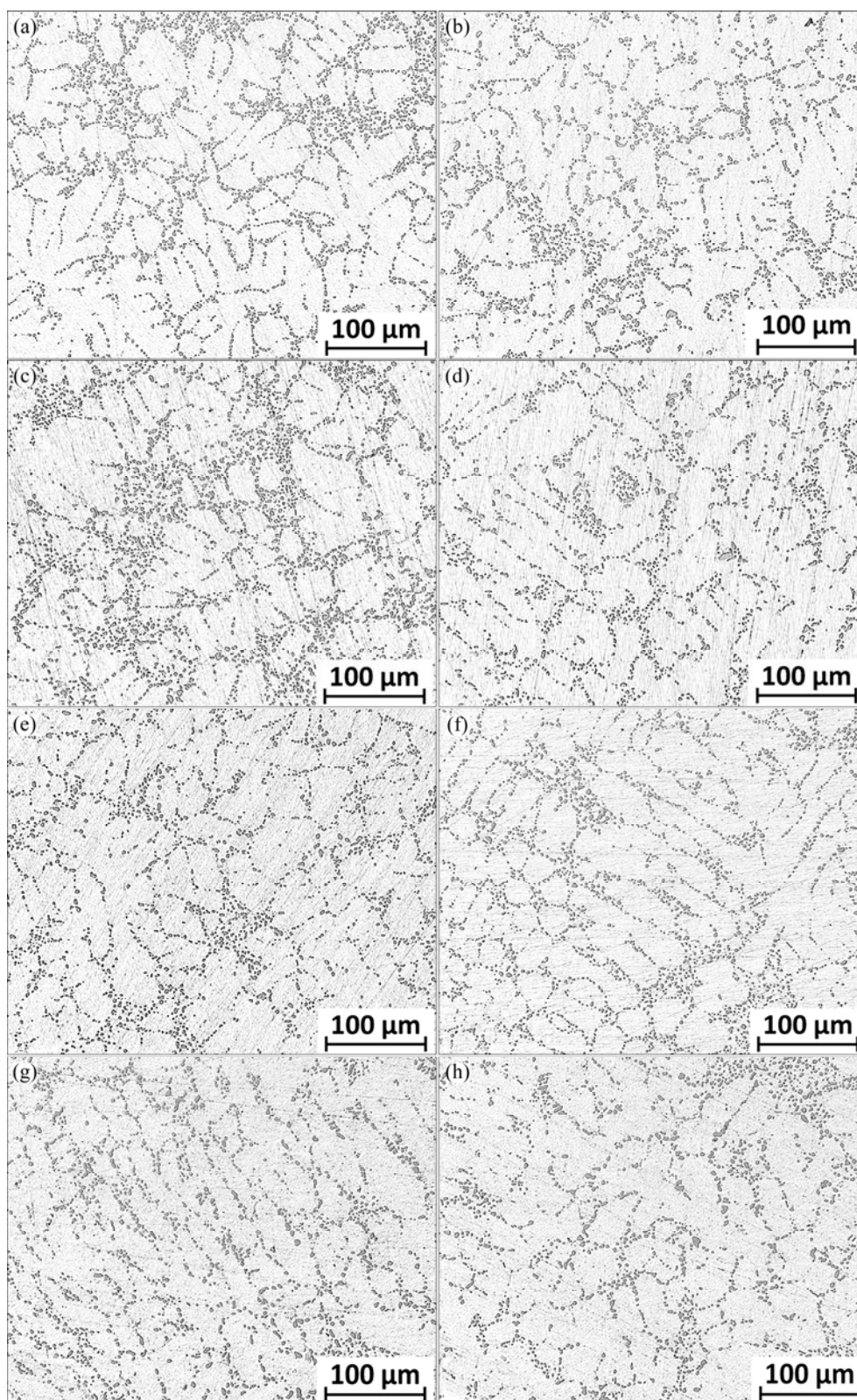


Fig. 7 Microstructures of B356 alloy aged at 155 °C for various time: (a) 2 h; (b) 3 h; (c) 4 h; (d) 5 h; (e) 6 h; (f) 8 h; (g) 16 h; (h) 32 h

precipitation of β'' phase during artificial aging. These effects confirm that β'' phase can be considered the main strengthening phase in the Al–Si–Mg alloys [3,7,12]. In

particular, the mechanical properties reach their maximum values when β'' phase is completely formed during artificial aging. According to DSC curves, this

happens when the peaks associated with β'' phase completely disappear, that is, this happens after 16 h for samples aged at 155 °C, after 6 h for samples aged at 165 °C and after 4 h for samples aged at 180 °C. The subsequent decrease of the mechanical properties observed in the sample aged at 180 °C with increasing aging time can be associated with the transformation of the coherent β'' phase into the semi-coherent β' phase, as also found in Refs. [7,11].

4 Conclusions

The age hardening behavior of a foundry alloy, namely the gravity cast B356 aluminum alloy, was studied. The influences of artificial aging temperature and time on the precipitation sequence of the alloy were investigated by DSC. The effects of the aging condition on mechanical properties of the alloy were evaluated through hardness and tensile tests. Afterwards, the mechanical properties were correlated with the DSC results. It is found that they reach the maximum values when β'' phase is completely formed: this happens after 16 h for samples aged at 155 °C, after 6 h for samples aged at 165 °C and after 4 h for samples aged at 180 °C. Since the activation energy is an important parameter in the quantification of alloy age hardening behaviour, this parameter was calculated by DSC curves and a value of 57.2 kJ/mol was found.

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人工时效条件对重力铸造 B356 铝合金力学性能的影响

D. DIONI, S. CECCHEL, G. CORNACCHIA, M. FACCOLI, A. PANVINI

Department of Mechanical and Industrial Engineering, University of Brescia, Via Branze 38-25123 Brescia, Italy

摘 要: 采用差示扫描热量法(DSC)、硬度测试和拉伸试验研究重力铸造 B356 铝合金的时效硬化行为。人工时效温度分别为 155、165 和 180 °C, 热处理时间为 40 min~32 h。DSC 结果表明, 样品在低于室温的条件下(约为 -10 °C)形成团簇。由于团簇形成影响主要强化相 β'' 的后续沉淀过程, 因此可以推断, 固溶处理后不及时进行人工时效处理将对合金的力学性能产生不利影响。同时证实, 在人工时效过程中(分别在 155 °C 时效 16 h, 165 °C 时效 6 h 和 180 °C 时效 4 h)当 β'' 相生成完全后, 合金的硬度和拉伸强度达到最大。经最高时效温度处理的样品其力学性能随时效时间的延长而降低, 这与共格 β'' 相向半共格 β' 相的转变相关。最后计算得到与 β'' 相沉淀相关的活化能为 57.2 kJ/mol。

关键词: B356 铝合金; 重力铸造; 人工时效; 析出序列; 力学性能

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