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Simultaneous hot isostatic pressing and solution annealing of aluminum cast alloys followed by instantaneous aging at elevated temperatures

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Abstract. Age-hardenable aluminum - silicon - magnesium cast alloys are widely applied in automotive and aircraft industries. Hot isostatic pressing is a common method to increase the materials fatigue resistance by reducing the inner porosity of aluminum cast materials. In the presently followed industrial production route hot isostatic pressing is performed preceding the standard heat treatment consisting of solution annealing and aging. In previous work Hafenstein et. al. demonstrated the possibility to achieve an oversaturated condition with a sufficient amount of magnesium and silicon atoms dissolved in the aluminum matrix by performing rapid temperature changes in advanced hot isostatic presses. Hence, it becomes possible to combine hot isostatic pressing and solution annealing within a single process step and to perform aging at elevated temperatures immediately after hot isostatic pressing without the necessity of separate solution annealing. The present study evidences that the design of a fully integrated process which comprises not only hot isostatic pressing and solution annealing but also aging at elevated temperatures is possible thereby shortening further the overall processing time. It is shown that the benefit with respect to strength in such a combined process is due to the shortening of the time at room temperature between solution annealing and aging at elevated temperatures.

1. Introduction

Pores are major sites for the initiation of fatigue cracks in aluminum cast material. Due to thermally activated dislocation creep and diffusion creep processes, inner porosity of aluminum cast material can be reduced by hot isostatic pressing (HIP) [1–4]. Hot isostatic pressing is conventionally done preceding to the regular heat treatment, which is composed of solution annealing, quenching and aging. This ensures high and reproducible component quality and meets the high demands on fatigue resistance for applications in the automotive and aircraft industries [5]. The high strength of age-hardened aluminum cast alloys is achieved by solution annealing, followed by quenching and aging at elevated temperatures which is performed within two separate process steps following hot isostatic pressing [6]. An oversaturated condition of magnesium and silicon atoms dissolved in the aluminum matrix is required in order to form a disperse distribution of various precipitates which increase the materials strength during artificial aging [7–10]. Magnesium and silicon atoms will remain as substitutional alloying elements within the aluminum matrix and form an oversaturated condition if the quenching rate after solution annealing is high enough to avoid diffusion during quenching. Due to the limited capability

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to perform rapid temperature changes within a standard hot isostatic press, densification, homogenization and quenching cannot be performed within a single step [11, 12]. Conducting three separate process steps is a significant cost factor opposing a broad industrial exploitation of such production routes. It has been shown that an increase of the cooling rate after hot isostatic pressing can ensure a sufficient amount of magnesium and silicon atoms dissolved in the aluminum matrix to achieve age-hardenability of the aluminum cast material [13,14]. Therefore, the costs of a production route which includes hot isostatic pressing could be possibly reduced by combining hot isostatic pressing and solution annealing in a single process step [13]. It is shown that instantaneous aging after hot isostatic pressing offers benefits with respect to strength by avoiding pre-aging at room temperature as is the case in standard heat treatment routes. The aim of the present study is to investigate the possibility of a combined process comprising hot isostatic pressing, solution annealing and aging. The findings of this work include results of mechanical testings and microstructural analysis performed on hot isostatically pressed samples of the aluminum cast alloy AlSi7Mg0.3 (A356).

2. Experimental

2.1. Casting

Samples of the alloy AlSi7Mg0.3 were produced via sand casting by Georg Fischer AG, Schaffhausen. Figure 1 shows a schematic illustration of the cast samples. The melt was degassed and refined by the addition of strontium. All alloying elements were added to the melt at a temperature of 730 °C. The chemical composition of the Alloy A356 is defined in the standard *EN 1706:2010* [15]. The measured composition is given in Table 1 and was analyzed by spark emission spectroscopy using a SPECTROMAXx LMM16 spark emission spectrometer (Spectro, Germany). The melt was exposed to a gassing procedure in order to achieve an enhanced casting porosity of $V_{\mathrm{P}} = 0{,}17\,\%$. Heat treatment and hot isostatic pressing was performed on cylinders possessing a diameter of 16 mm and a length of 70 mm which were machined from the cast material.

Table 1. Chemical analysis of the aluminum cast alloy AlSi7Mg0.3

Element	wt.-%		Deviation wt.-%
Si	7.323	±	0.008
Fe	0.125	±	0.002
Cu	0.022	±	0.001
Mn	0.029	±	0.001
Mg	0.345	±	0.001
Ni	0.005	±	0.001
Zn	0.088	±	0.072
Ti	0.125	±	0.003
Al	balance		

2.2. HIP process and heat treatment

The mechanical properties of the alloy AlSi7Mg0.3 were determined in as-cast and various hot isostatically pressed conditions. Samples of the series *HIP 540°C 2h -0.2K/s* and *HIP 540°C 2h - 0.2K/s -A* were subjected to conventional hot isostatic pressing at 540 °C for 120 min without subsequent solution annealing. The quenching rate within the conventional HIP process was

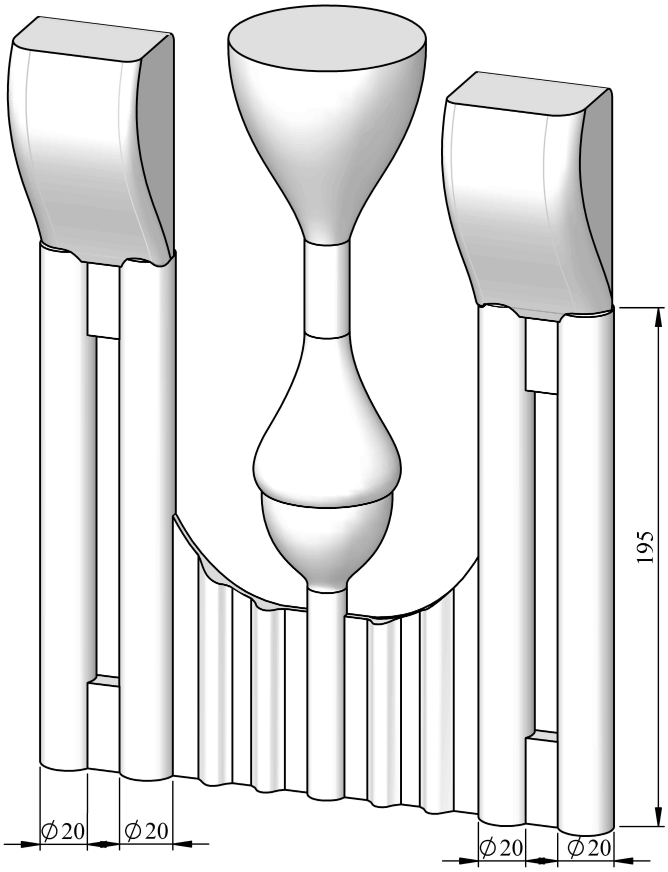

Figure 1. Schematic illustration of the samples produced by sand casting; units in mm

about 0,2 K/s within the temperature range between 540 and 200 °C. Samples of the conditions *HIP 540°C 2h -0.2K/s* were hot isostatically pressed and aged for more than seven days at room temperature. Samples of the hot isostatically pressed condition *HIP 540°C 2h -0.2K/s -A* were also pre-aged at room temperature and then aged at 165 °C for 150 min. The temperature over time profile of the two heat treatment conditions is shown in Figure 2. Samples of the heat treatment conditions *HIP 540°C 2h -7K/s* and *HIP 540°C 2h -7K/s -A* were hot isostatically pressed at 540 °C for 120 min and quenched with a rate of 7 K/s. Samples of the conditions *HIP 540°C 2h -7K/s* were hot isostatically pressed and aged for more than seven days at room temperature. Samples of the hot isostatically pressed condition *HIP 540°C 2h -7K/s -A* were additionally aged at 165 °C for 150 min. The temperature over time profile of the two heat treatment conditions *HIP 540°C 2h -7K/s* and *HIP 540°C 2h -7K/s -A* is shown in Figure 3. Samples of the heat treatment conditions *HIP 540°C 2h -7K/s -DA* were hot isostatically pressed at 540 °C for 120 min, quenched with a rate of 7 K/s after hot isostatic pressing and then instantaneously aged at 165 °C for 150 min. The temperature over time profile of this direct-age (DA) condition is shown in Figure 4.

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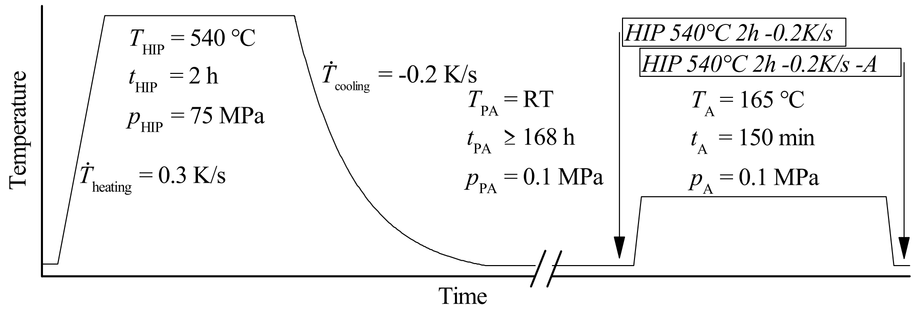

Figure 2. Temperature over time profile for the heat treatment conditions *HIP 540°C 2h - 0.2K/s* and *HIP 540°C 2h -0.2K/s -A*.

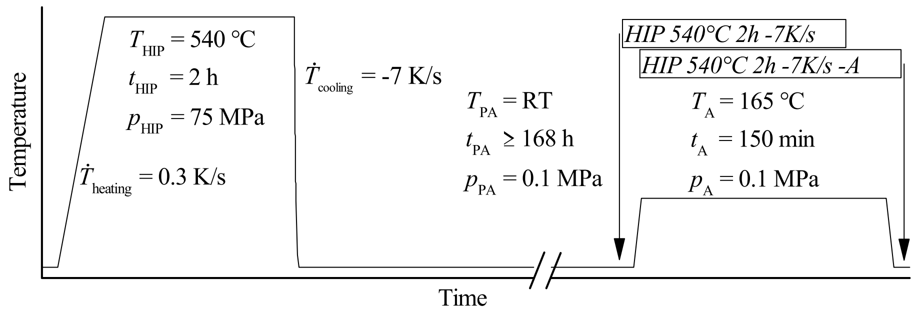

Figure 3. Temperature over time profile for the direct-age heat treatment condition *HIP 540°C-2h -7K/s* and *HIP 540°C 2h -7K/s -A*.

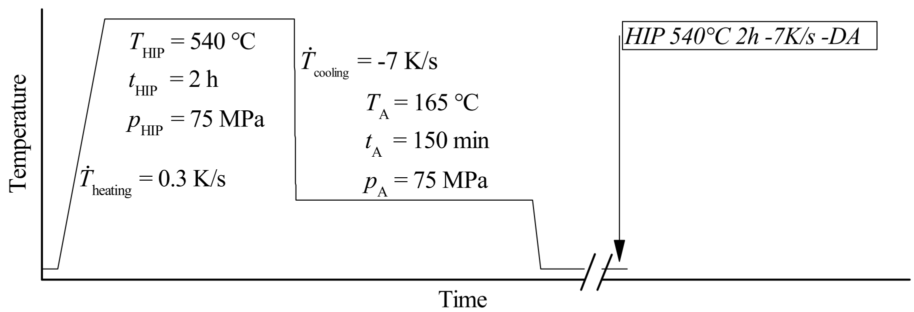

Figure 4. Temperature over time profile of the heat treatment conditions *HIP 540°C 2h -7K/s - DA*.

2.3. Mechanical testing

The sample shape was chosen according to DIN 50125:2004-01, type B, size M10 [16]. Tensile testing was carried out on an Instron 4505 tensile testing machine connected to a computer using the operating software Bluehill 2. An extensometer was used to measure the strain parallel to the cylinder axis. The tests were performed at a strain rate of $\dot{\varepsilon} = 10^{-4}\,\mathrm{s}^{-1}$. Round fatigue test samples, with dimensions as shown in Figure 5, were machined from the center of the heat treated cylinders. Fatigue resistance testing was carried out on a RUMUL high frequency resonant testing machine (Russenberger Prüfmaschinen AG, Switzerland). Due to the limited capacity of the HIP vessel, the fatigue resistance of the hot isostatic pressed conditions was compared at a single alternating stress level (R = -1, $\sigma_\mathrm{m} = 0\,\mathrm{MPa}$). The maximum amplitude was set at $\sigma_\mathrm{a} = 75\,\mathrm{MPa}$.

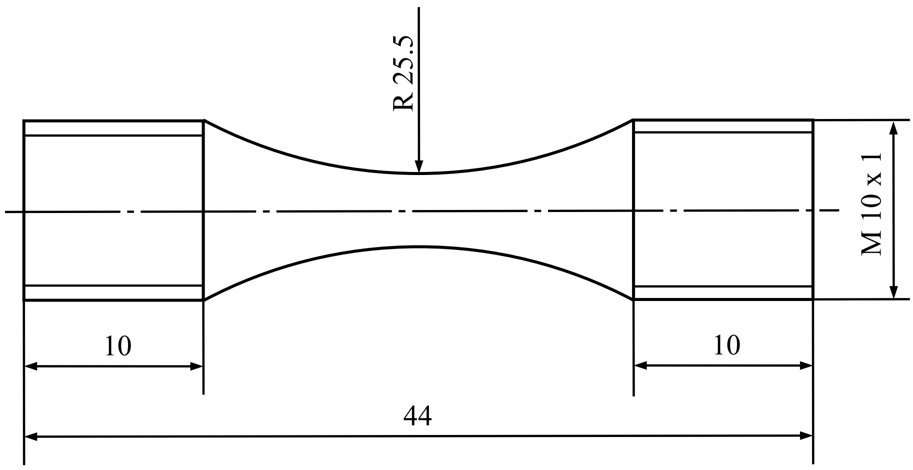

Figure 5. Drawing of the samples used for fatigue testing. Units in mm.

Hardness measurements where performed according to DIN EN ISO 6506-2 using an M4U-025 hardness testing machine (EMCO-TEST, Austria) [17].

3. Results and Discussion

Porosity affects different material properties with various intensities [18]. It is known that the porosity has a minor effect on the material's yield stress and hardness when compared to its effect on ultimate tensile strength, elongation at fracture and fatigue resistance [19, 20]. The fatigue resistance and the elongation at fracture of the *as-cast* sample series were found to be the lowest of all investigated conditions, whereas hardness and yield stress where at least as high as higher level then the ones of the conventionally hot isostatically pressed conditions *HIP 540°C-2h-0.2K/s* and *HIP 540°C 2h-0.2K/s-A*. This indicates that the quenching rate during sand casting is the same or even higher when compared to the standard HIP treatment. The preferred starting point for crack initiation is the interface between the matrix and large precipitates or pores inside the material [2, 21–24]. The number of cycles for crack initiation in fatigue resistance tests is significantly larger than the number of cycles during the growth of these cracks. The low fatigue resistance of the *as-cast* sample series is therefore a result of the high number of preferred starting points for crack initialization. It can be noted, that the fatigue resistance and the elongation at fracture is significantly increased by hot isostatic pressing even thought the yield stress, the hardness and the ultimate tensile strength of the conventionally hot isostatically pressed conditions *HIP 540°C 2h-0.2K/s* and *HIP 540°C 2h-0.2K/s-A* are as low as for the *as-cast* condition. The reason for the low yield stress, hardness and ultimate tensile strength of the conventionally hot isostatically pressed conditions can be found in the microstructural properties of these conditions, which are influenced by the low cooling rate of 0.2 K/s in the conventionally hot isostatic pressing process and the resulting distribution of alloying elements. A low cooling rate goes along with long exposure times at high temperatures, which results in stable and coarse precipitates of the alloying elements silicon and magnesium formed at elevated temperatures [25]. Therefore there are not enough silicon and magnesium atoms in solid solution which could form needle-like β'' precipitates during artificial aging that are known to be the main cause of strength improvement [26, 27]. These results indicate that the quenching rate of 0.2 K/s, following densification in the conventional hot isostatic pressing process is not high enough to produce a suitable oversaturated solid solution. The application of a second heat treatment step dedicated to artificial aging at 165 °C for 150 min leads to a slight increase of the properties yield stress, hardness, ultimate tensile strength, but causes lower values

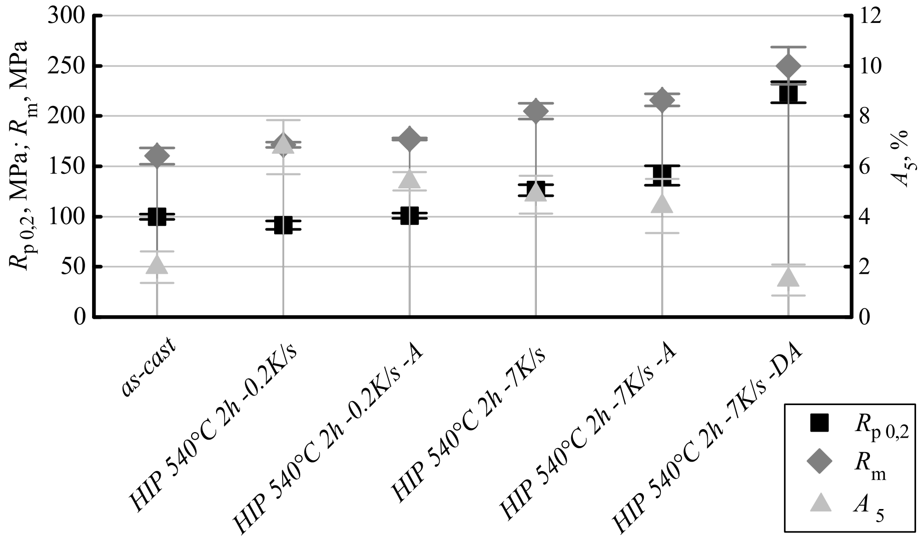

Figure 6. Tensile test results ($R_{p\,0,2}$, R_m and A_5) of all investigated samples corresponding to the performed thermal treatment; the error bar represents the standard deviation between three conducted measurements per heart treatment condition.

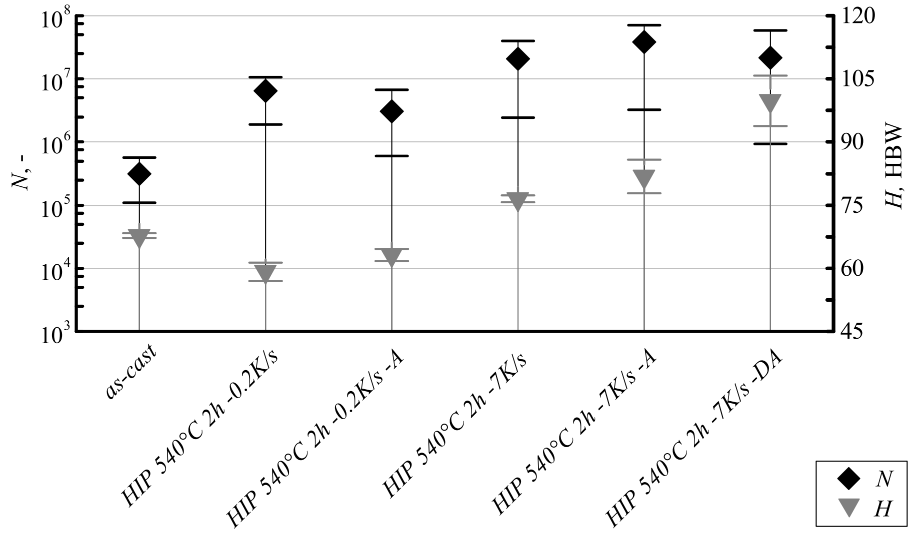

Figure 7. Cycles until failure (N) and Brinell Hardness (H) of all investigated samples corresponding to the performed thermal treatment; the error bar represents the standard deviation between three conducted measurements per heart treatment condition.

for the elongation at fracture.

With an increase of the cooing rate after hot isostatic pressing from 0,2 to 7 K/s, the yield stress of the conditions aged at room temperate (*HIP 540°C 2h -0.2K/s* and *HIP 540°C 2h -7K/s*) is increased by 38 % from 91 to 126 MPa. Concurrently the ultimate tensile strength increases by 19 % from 172 to 204 MPa and hardness by 31 % from 59 to 77 HBW. The high strength of the *HIP 540°C 2h -7K/s* heat treatment condition causes a significant higher fatigue resistance and a lower elongation at fracture when compared to the conventional condition *HIP 540°C 2h -0.2K/s*. After artificial aging at 165 °C for 150 min the heat treatment condition *HIP 540°C 2h -7K/s -A* exhibits a yield stress of 140 MPa, an ultimate tensile strength of 116 MPa and a hardness of

82 HBW. Therefore the strength level as well as the fatigue resistance of the artificially aged condition is higher than they are after room temperature aging, whereas elongation at fracture is slightly lower. It has been demonstrated previously that a quenching rate of 7 K/s after hot isostatic pressing is high enough to archive an oversaturated solid solution and enables aging to be performed after hot isostatic pressing without the necessity of separate solution annealing [13,14]. Artificial aging at 165 °C for 150 min was applied directly after hot isostatic pressing for the heat treatment condition *HIP 540°C 2h - 7K/s - DA* such samples exhibit the highest mechanical properties. When compared to the heat treatment condition *HIP 540°C 2h - 7K/s - A* which was artificially aged after one week of pre-aging at room temperature but with the same aging parameters, the heat treatment condition *HIP 540°C 2h - 7K/s - DA* shows superior mechanical properties. The yield stress of the directly aged condition *HIP 540°C 2h - 7K/s - DA* is 224 MPa and therefore 58 % higher than the yield stress of the heat treatment condition *HIP 540°C 2h - 7K/s - A*. Ultimate tensile strength and hardness of this direct-age condition are 250 MPa and 100 HBW, and respectively, are also significantly higher than that of the pre-aged samples series *HIP 540°C 2h - 7K/s - A*. The fatigue resistance of *HIP 540°C 2h - 7K/s - DA* is at the same level as that of *HIP 540°C 2h - 7K/s - A*. The elongation at fracture of the directly aged condition is found to be the lowest of all investigated sample series.

The influence of pre-aging at room temperature on the resulting mechanical properties of an aluminum alloy has been subject of previous investigations [28–30]. Short instantaneous aging of 5 min at 150 °C can reduce the detrimental effect of room temperature pre-aging on the resulting mechanical properties [31]. It has been shown hat the influence of pre-aging depends on the alloying composition as well as on the duration and the temperature [32–36]. The detrimental effect is based on the clustering of silicon and magnesium atoms during pre-aging [37]. The composition of the clusters formed in the early stage of the precipitation sequence depends on the temperature during their formation. The composition of the clusters affects the resulting mechanical properties of the alloy by promoting or restraining the formation of strength improving β'' precipitates during artificial aging which are the main source of strength improvement [38, 39].

4. Summary and Outlook

Quenching rates of 7 K/s within the temperature range between 540 and 200 °C are high enough to archive a suitable oversaturated solid solution after hot isostatic pressing. A fully integrated process which comprises not only hot isostatic pressing and solution annealing but also aging at elevated temperatures shortens the overall process time. Due to the clustering of silicon and magnesium atoms during room temperature pre-aging, a combined process with immediate aging at elevated temperatures possesses benefits with respect to strength. Taking into account the outstanding mechanical properties achieved by direct aging at elevated temperatures, even lower cooling rates than 7 K/s could be high enough to archive adequate material strength.

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