The Structure and Matrix Microhardness of the 319 Aluminum Alloy After Isothermal Holding During the Solidification Process

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ABSTRACT

The 319 aluminum alloy is used in a variety of automotive applications. This alloy is typically solution treated in order to improve its metallurgical and mechanical properties. "Traditional" solution treatment involves cooling the castings to an ambient temperature before heating them to the solution temperature; however, some foundries have attempted to institute a more efficient procedure by shortening or eliminating the cooling step.

The purpose of this paper is to investigate the effect of isothermal holding during the solidification process on the structure and matrix microhardness of 319 aluminum alloy test samples. The investigation reveals that isothermal holding at approximately 520°C minimizes the volume fraction of Cu enriched phases while maximizing the Vickers microhardness of the aluminum matrix. This suggests that 520°C is the ideal temperature for solutionizing Cu enriched phases in this alloy. Thermal analysis data from selected experiments are presented for illustrative purposes.

This paper represents the first in a series of investigations comparing the properties of castings solution treated using "traditional" methods and those subjected to isothermal holding during the solidification process.

INTRODUCTION

The 319 aluminum alloy is commonly used in the automotive industry as a material for engine blocks and cylinder heads. In order to improve the mechanical properties of such components they are often heat treated (HT) using a two step process (i.e. solution treatment (ST) and artificial aging (AA)). ST can be combined with thermal sand removal (TSR) as one continuous operation.

Two important ST process variables are time and temperature. They are responsible for Si particle modification and the dissolution of Cu and Mg enriched phases (a process that strengthens the alloy matrix). The conventional ST temperature must be kept below the melting point of the Cu enriched phases in order to avoid incipient melting; however, it should be high enough to create the optimum conditions for Cu enriched phase dissolution and Si sphereoidization (Gauthier et al., 1994; Sokolowski, et al., in press; Sokolowski et al., 1995). The concentration of alloying elements like Cu and Mg should also be optimized in order to obtain the maximum solid solution strengthening effect and to maintain the properties of the alloy under elevated temperature conditions (ASM Specialty Handbook, 1993; Aluminum: Properties and Physical Metallurgy, 1984). An excess concentration of alloying elements and impurities creates macrosegregation, which is harmful to mechanical properties.

The behaviour of the 319 alloy during isothermal holding is worthy of investigation because it is frequently encountered during foundry operation and it may have an effect on casting mechanical properties. Many foundries have attempted to decrease the time between pouring and TSR by holding the semi-solid casting at the TSR temperature rather than allowing it to cool to the ambient temperature and then initiating TSR. Besides making the casting operation shorter, this procedure saves energy; which makes it economically advantageous.

The response of the 319 alloy during isothermal holding is poorly documented in the literature. Most practitioners assume that as long as solution treatment temperatures and times are kept constant the substitution of isothermal holding for
"traditional" procedures should have little effect on casting properties. However, preliminary laboratory investigations conducted by the authors (Kasprzak, et al., 2000) have revealed an unexpected increase in the 319 alloy matrix microhardness during isothermal holding, especially when the Cu enriched phase reaction temperature is approached. The microhardness of samples isothermally held at 500°C for 90 minutes was approximately 120 HV25. This is about 20% higher when compared with samples heated from the ambient temperature to a temperature close to the Cu enriched phase incipient melting point (about 500°C), held for 90 minutes and cooled in still air. Moreover, the area fraction of the Cu enriched phases in the isothermally held samples was approximately 0.4%, which is about 30% smaller than those found in the "traditionally" heat treated sample (see Figure 1).

The observed differences in response to the two different heat treatment cycles establish the need for investigation of isothermal holding on casting properties and a comparison with "traditional" heat treatment techniques. The purpose of this paper is to present the influence of isothermal holding during solidification on the structure and matrix microhardness of 319 aluminum alloy samples.

MATERIALS AND EXPERIMENTS

The experiments were carried out using cylindrical specimens machined from a 319 alloy casting; the chemical composition is presented in Table 1:

Table 1. Chemical composition of the 319 aluminum alloy.

<table>
<thead>
<tr>
<th>Alloy type</th>
<th>Average composition, wt %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Si</td>
</tr>
<tr>
<td>319</td>
<td>7.70</td>
</tr>
</tbody>
</table>

Fig. 1. The area fraction of Cu enriched phases (AFCu%) and matrix microhardness (HV25) for samples:
- "Traditionally" solution treated at 500°C for 90 min and cooled in still air,
- Isothermally held at 500°C for 90 min during the solidification process and cooled in still air,
- In the as-cast condition.
In order to determine which metallurgical reactions took place during the experiments, thermal analysis was performed. A specially designated, supersensitive K-type thermocouple (with an extra low time constant) was placed 7mm from the top of each cylindrical sample (diameter = 16mm, length = 15mm, mass = 8.2g) and connected to a high speed National Instruments data acquisition system. The sample was wrapped with a 25µm thick brass sheet, placed on a ceramic stand and covered with a ceramic cap. It was heated to a liquid state (~750°C) inside a high frequency resonant inverter developed at the Silesian University of Technology (Kasprzak, 1997), using a constant power input and allowed to cool to the ambient temperature after the power was turned off. The temperature and its first derivative vs. time curves were plotted. This procedure was repeated for subsequent samples, except that they were held at holding temperatures between 540 and 470°C (with an accuracy of +/-0.5°C) for periods of 90 minutes before being allowed to cool to the ambient temperature.

![Graph showing heating and cooling curves and first derivative curve for the 319 alloy samples.](image)

**Fig. 2. Heating and cooling curves and first derivative curve for the 319 alloy samples.**

*Note the main metallurgical reactions during heating and solidification:*

1. dissolution of Cu enriched phases,
2. dissolution of Al-Si eutectic,
3. dissolution of Al dendrite network,
4. nucleation of Al dendrite network - 598.4°C,
5. nucleation of Al-Si eutectic - 562.7°C,
6. nucleation of Cu enriched phases - 495.3°C.

Samples for metallographic observations and matrix microhardness measurements were ground with abrasive paper and polished using BUEHLER company equipment. The final polishing was carried out using commercial 0.05µm slurry. The area fraction of the Cu enriched phases in the samples was evaluated using a LEICA QW-550 image analysis system linked to a Leica DMR light optical microscope. Twenty-five (25) analytical fields were measured under 500x magnification for each sample and the median Cu enriched phase volume fraction was determined.
Additional metallographic observations were made using a JEOL JSM 5800 Scanning Electron Microscope (SEM) in Back Scattered Electron (BSE) mode under magnifications varying between 100 and 2000x. The matrix Vickers microhardness was measured using a BUEHLER microhardness tester under a 25g load for 15s (HV25). Twenty measurements were taken per sample and the median microhardness was determined. Microhardness measurements were performed after two weeks so that the material could naturally age. Matrix microhardness measurements were chosen in place of overall macrohardness measurements to evaluate the dissolution of Cu enriched phases and other structural constituents on the solid solution strengthening effect.

RESULTS AND DISCUSSION

Thermal analysis revealed that three main metallurgical reactions can be recognized during the solidification process: the nucleation of the Al dendrite network @ 598.4°C, nucleation of the Al-Si eutectic phase @ 562.7°C and the nucleation of the Cu enriched phases @ 495.3°C (Figures 2 and 2a). Three convoluted peaks can be recognized on the first derivative curve which pertain to the Cu enriched phase reaction (Djurdjevic et al., 2000). The cooling rate, calculated between T_{liquidus} and T_{solidus}, was 0.76°C/s. This relatively high rate can be attributed to the small sample mass.

Observation of the as-cast sample under a Light Optical Microscope (LOM) showed a typical unmodified microstructure.

![First derivative curve vs. temperature during cooling and heating for the 319 alloy samples.](image)

*Fig. 2a. First derivative curve vs. temperature during cooling and heating for the 319 alloy samples.*

*Note the main metallurgical reactions during heating and solidification:*

1 - dissolution of Cu enriched phases,
2 - dissolution of Al-Si eutectic,
3 - dissolution of Al dendrite network,
4 - nucleation of Al dendrite network - 598.4°C,
5 - nucleation of Al-Si eutectic - 562.7°C,
6 - nucleation of Cu enriched eutectic – 495.3°C.

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1 The median was chosen as the criterion of analysis in this study because it is less influenced by extreme scores, the presence of which is a concern in both the measurement of microhardness and Cu enriched phase area fraction.
consisting of the aluminum matrix, Al-Si and Al-Cu eutectics and Fe enriched phases in the form of Chinese script and needle like formations (Bäckerud et al., 1986) (Figure 3). SEM observation confirms the earlier assertion that Cu enriched phases appeared in three main morphologies: blocky, eutectic type and fine eutectic type (Djurdjevic et al., 2000). Mg enriched phases (Mg2Si) was also observed under the SEM.

Measurements of the Cu enriched phase area fractions showed that isothermal holding of the test sample during solidification decreased the area fraction when compared with the as-cast sample (area fraction ~ 2.6%). Increasing the holding temperature from 470 to 520°C and holding for 90 minutes decreased the area fraction of the Cu enriched phases from about 2.4 to 0.2%. Isothermal holding at 530 and 540°C caused an increase in the area fraction of the Cu enriched phases to approximately 0.5% to 1.2%, respectively (Figure 4).

The change in the Cu enriched phase area fraction between the as-cast sample and the sample held at 510°C (which has a Cu enriched phase area fraction of about 0.3%) is shown in Figures 5 and 6. The smallest Cu enriched phase area fraction after isothermal holding occurred in the temperature range between 500 and 530°C. Additional statistical analysis of the experimental data revealed that the differences in area fractions for the samples isothermally held between 500 to 530°C are not significant (probability > 0.05).

The matrix microhardness measurements showed that isothermal holding during solidification increased the microhardness of the Al matrix compared to the as-cast material (HV25 = 78). An increase in holding temperature from 470 to 520°C increased the matrix microhardness from about 90 to 122 HV25. A significant decrease in microhardness to about 102 HV25 was observed when the holding temperature exceeded 540°C (Figure 7). The maximum matrix microhardness of approximately 120-122 HV25 was reached between 500 and 520°C. Statistical analysis revealed that the microhardness differences in this temperature range are not significant (probability > 0.05).
Fig. 4. Area fractions of the Cu enriched phases for the samples isothermally held at various temperatures for 90 minutes during the solidification process. As-cast material is included in the chart as a reference.

Fig. 5. SEM micrograph (BSE image) of the as-cast sample. The Cu enriched phases appear brightest #1. Fe enriched phases appear as “chinese script” and are darker gray #2.
Fig. 6. SEM micrograph (BSE image) of the sample isothermally held at 510°C for 90 minutes during the solidification process. The Cu enriched phases appear brightest #1. Fe enriched phases appear as “chinese script” and are darker gray #2. Note the considerable decrease in the area fraction of the Cu enriched phases compared to the as-cast structure (Figure 5).

Fig. 7. Vickers microhardness measurements (HV25) of the aluminum matrix for the samples isothermally held at various temperatures for 90 minutes during the solidification process. As-cast material is included in the chart as a reference.
The changes in the matrix microhardness are correlated with the area fractions of the Cu enriched phases. Figures 4 and 7 show that the maximum microhardness of approximately 120-122 HV25 corresponds to the minimum area fraction of the Cu enriched phases (i.e. 0.2-0.4%) which occurs after holding at between 500 and 520°C.

Several factors must be taken into consideration to explain these findings. The dissolution of Cu enriched phases during isothermal holding increases the concentration of Cu and other alloying elements (Mg, Si) in the aluminum matrix. Cu and Mg are considered primary solid solution strengthening agents and their effect is clearly reflected by the HV25 matrix measurements. Cu and Mg also create dispersed intermetallic precipitates and increase the overall matrix strength by a mechanism called the precipitation strengthening effect (Aluminum: Properties and Physical Metallurgy, 1984; ASM Specialty Handbook, 1993). Unfortunately, because these precipitates are very fine, their area fractions cannot be quantified using image analysis combined with light optical microscopy.

The relationship between holding temperature, the area fraction of the Cu enriched phases and the matrix microhardness is complex. To clarify the metallurgical reactions that occur during isothermal holding the application of more sophisticated analytical techniques is required (e.g., Transmission Electron Microscopy observations combined with X-ray spot microanalysis of quenched specimens).

With respect to the thermal analysis data, a comparison of the first derivative vs. temperature plots for the as-cast sample
Figure 8) to those isothermally held at 530°C (Figure 9) and 520°C (Figure 10) showed a decrease in the “energy signature” of the Cu enriched phase reaction in the held samples. It is plausible that the metallurgical reaction energy signature can be estimated using the area between the first derivative curve and the base line curve. This hypothesis is investigated in a subsequent paper (Djurdjevic et al., 2000).

It is possible that this reduced metallurgical reaction “energy signature” can be attributed to the fact that the diffusion of alloying elements to the Al matrix becomes more effective when the nucleation temperature of the Cu enriched phase reaction is approached. That is, more Cu and Mg are able to diffuse into the matrix rather than taking part in the formation of Cu enriched phases. Consequently, the “volume” of liquid which undergoes a transformation (from liquid to solid state) is smaller when compared with a sample solidified without isothermal holding or solidified with isothermal holding below the temperature of the Cu enriched phase reaction.

CONCLUSIONS

For the investigated alloy, an isothermal holding temperature of between 500 and 520°C appears to be optimal. This temperature range allows the majority of the Cu enriched phases to go into solution which in turn results in the maximum matrix microhardness (HV25 ~ 120). Higher temperature isothermal holding results in a decrease in the matrix microhardness (HV25 = 109 @ 530°C) while lower temperature holding does not maximize the dissolution of the Cu enriched phases.

**Fig. 9. First derivative vs. temperature of the cooling curve for 319 alloy isothermally held at 530°C for 90 minutes during the solidification process. The holding temperature was 35°C above the nucleation of the Cu enriched phases, e.g., ~495°C. The holding temperature is marked by an arrow. Note the main metallurgical reactions during the solidification process: 1 - nucleation of Al dendrite network, 2 - nucleation of Al-Si eutectic, 3 - nucleation of Cu enriched phases. Note the smaller “energy signature” of the Cu enriched phase reaction compared with the as-cast material (Figure 8).**
Future work by the authors will address the following issues:

1. Is there a significant difference between "traditional" heat treatment and isothermal holding during the solidification process in terms of the structures produced and the metallurgical properties of the resulting castings?
2. What is the precise effect of isothermal holding during the solidification process on phase formation?

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REFERENCES