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Influence of Gas Oven Temperature on the Microstructure and Extrudability of Al-Mg-Si Aluminium Alloys

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Abstract. The microstructure of Al-Mg-Si aluminium alloys is well characterised when it comes to the initial state of the cast billet or the artificially aged profile. Typically, a gas oven coupled with an induction oven is used to preheat the billets before extrusion. Preheating reduces the flow stress of the aluminium and dissolves the phases that have precipitated during cooling after homogenisation.

In this paper, the influence of the gas oven temperature on the partial or complete dissolution of the precipitated phases will be shown. In addition, the negative effect of a wrong choice of parameters on the volume content of the MgSi phases will be shown.

Introduction

The automotive industry is the main driving force for our developments. On the one hand, the mechanical properties shall become better, the ductility coupled with high strength. This allows to design and build light weight cars in special light and save battery frames and housing. On the other hand, the total CO_2 consumption has come into the focus of the OEM's. The CO_2 consumption of an alloy depends mainly on the recycling content. The needed energy for remelting and cleaning is less than 10% of the energy to produce smelted aluminium [1]. The recycled alloys are as good as the smelted alloys. The small difference is hidden in the background, by comparing the wt% of the chemical elements and the distribution of these. The wt% content is mostly on the upper end of the specification and the distribution width is not so narrow compared to the smelted alloy.
Royset et al. [2] showed that small reductions of Mg and Si can enhance the extrusion speed. On the basis of these results, a narrow distribution of the alloying elements on the lower limit of the specification is desired.

To characterise this phenomenon, the break through pressure can work as a comparison parameter or the flow stress determined from a lab deformation test [3]. The assumption is that all MgSi phases are dissolved during the extrusion process. This assumption is true on condition that all parameters like alloy composition, homogenisation and extrusion parameters were set perfectly. Perfect extrusion parameters launch with the alloy composition, chosen well at the lower limit of the specification and the used casting equipment is state of the art. The homogenisation is done at the highest alloy dependent temperature and the soaking time is long enough to fulfill the transformation of the AlFeSi phases. The cooling gradient from homogenisation temperature should be the same as the reheating gradient to extrusion temperature. The extrusion speed is set to the maximum speed, shortly before generating surface tear caused by overheating of the profile during deformation. But under industrial process conditions this is not fulfilled exactly.

For instance, the alloy chemistry is chosen at the upper end of the alloy specification for Si and Mg to get high mechanical properties and a better machinability [1,5]. During solidification of the alloy this leads to more primary MgSi phases on the grain boundary or between the dendrite arms, shown in Fig. 1. Also, the microsegregation of Mg and Si in the grains is high.

If Fe, Mn and/or Cr are present in the alloy, a second group of phases will appear, the AlFe(Mn,Cr)Si phases (further designated as AlFeSi). These phases precipitate directly out of the

melt at the end of the solidification process [6]. The AlFeSi phases look like needles (low Fe content) or like Chinese characters at a higher Fe, Mn and Cr content [7].
A homogenisation treatment offers a solution for both problems. During homogenisation the transformation of the β AlFeSi into α AlFeSi takes place [8]. The bigger β AlFeSi transform into smaller α AlFeSi. This is favoured for the extrusion process, the α AlFeSi phases are more rounded compared to the needle shaped β AlFeSi phases in low alloyed 6XXX alloys, therefore, the deformation of the alloy without any surface issues is enhanced [9]. The cooling speed after the homogenisation has no influence on the transformation of the AlFeSi phases [10–12].
Additionally, the homogenisation temperature lower than the solidus and higher than the solvus line levels the microsegregation in the grains. Simultaneously, the primary MgSi phases at the grain boundary are dissolved. For the MgSi phases, the cooling rate down to room temperature plays an important role. If the cooling rate is low, the MgSi phases start to reprecipitate during the cooldown. Some bigger MgSi phases nucleate at the AlFeSi phases as needles. Even if there are some dispersoids present, they can act as nucleation sites and the MgSi phases precipitate comparatively smaller [13]. If the samples were water quenched, only the transformed AlFeSi phases are visible. There are so many different possibilities how the microstructure of a homogenised billet can look like depending on the temperature, time and cooling speed. Fig. 2 shows an example for a 6005(A) alloy homogenised at 570 °C for 4 hours and cooled with 500 K/h.

The characterisation of the homogenised state of the billet is common. Therefore, many billet quality specifications are out there. One of the topics is the transformation of the AlFeSi phases and another topic is the size of the MgSi phases. Billet quality specifications from different extrusion companies mention that the size of the MgSi phases should be less than 2 µm. One of the leading quality documents is the BQA (billet quality assessment) from Hydro. According to this document, 2 µm is a standard value. But the definition is not clear. Are the primary or secondary MgSi deemed? But for the metallurgical effect, re-melting or dissolution, only the size counts.

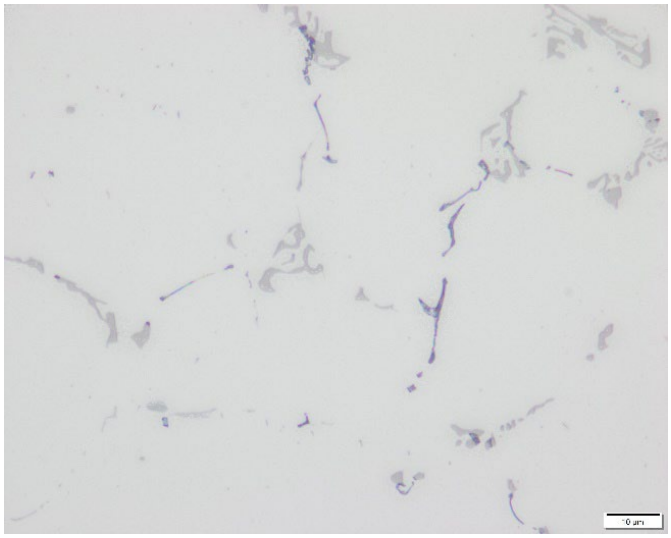


Fig. 1. Cast microstructure of a 6005(A) alloy, the AlFesi phases (grey) have a Chinese character structure. MgSi (blue, purple) is visible at the grain boundaries or the triple points. The measuring bar corresponds to 10 µm

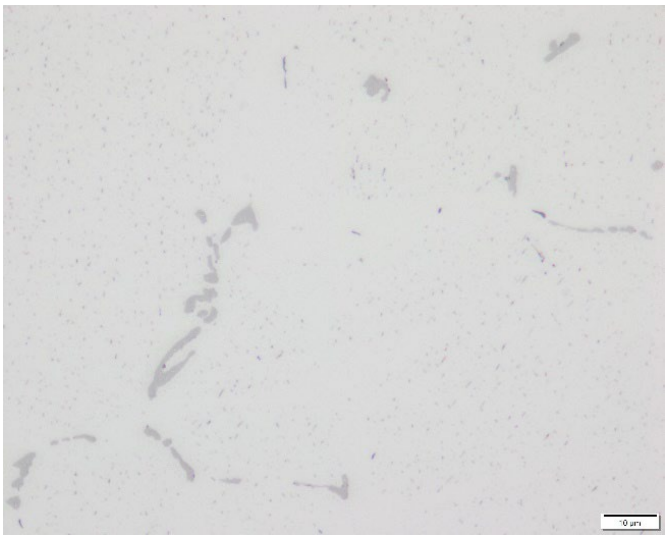


Fig. 2. The more rounded AlFeSi phases after homogenisation are visible. The small black dots in the grains are secondary MgSi phases. The big black ones on the grain boundary are the undissolved primary MgSi phases. The measuring bar corresponds to 10 µm

For the small MgSi phases it is postulated that they can re-dissolve during the extrusion process [7,14]. Fig. 3 shows the sketch of the extrusion process. The homogenised billet is reheated via a gas and induction oven to the start temperature for extrusion. After reheating, the MgSi phases should dissolve. Or as shown, the deformation and coupled increase of the temperature generates vacancies and, therefore, the MgSi phases have the ability to dissolve quickly. The highest break through pressure will be reached with all Mg and Si in solid solution. This is best practice if the extrusion press has enough power [14,15]. For this reason, less power of the press, one option is to increase the size of MgSi phases in the billet and try to dissolve the MgSi phases directly in front of the die. The billet is softer than in the state of a fully solid solution. When something went wrong with the extrusion parameters, some MgSi phases can survive and appear in the extruded profile. These phases are too big to enhance the mechanical properties. They tie up so much Mg and Si that the mechanical properties are dramatically decreased. Furthermore, big surviving MgSi phases can lead to local melting and a poor surface quality [16]. In the case of excessive surviving MgSi phases it could lead to hot tearing cracks. They look like melted grain boundaries. If all Mg and Si is in solid solution and the profile is cooled appropriately, it is capable of reaching the maximum strength, see Fig. 3. Therefore, the homogenisation treatment is mandatory before hot extrusion of 6XXX aluminium alloys [8,12,17,18].

It is assumed that during the reheating of the billets with the gas and induction oven the MgSi phases will dissolve. In the past, there was a big lack of knowledge with regard to what really happens in these process steps. The billets were hot sheared and transported to the press as soon as possible and loaded into the container. The accessibility to samples from the industrial environment is very low. Quenching of a hot sheared billet with approximately 200 kg is very risky and not a suitable way to get some proper homogeneous cooled samples. By adapting the gas oven and the hot saw placed directly behind, samples can be taken out of the process in a simple way. With the installation of the new gas oven line from extrutec, the problem was solved [19]. Slices with a thickness of 80 mm can be taken directly out of the process. The time between cutting and quenching is less than 20 seconds. During this short period there is no negative influence on the microstructure.

Samples were also produced on a laboratory scale because this is where the extreme tests can be carried out that are not possible on an industrial scale. But these tests are needed for the further development of the equipment. The samples obtained in that way will show that they differ from the expected results. The aim of this work is to investigate these microstructural dependencies of MgSi phases under consideration of the time and temperature regime of the gas and induction oven under industrial conditions. These approaches will help to explain some extrusion defects such as reduced mechanical properties or surface melting.

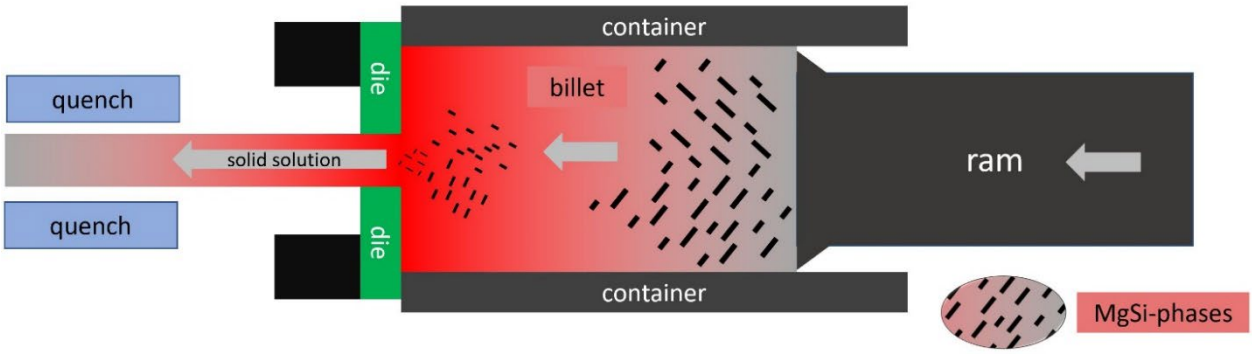


Fig. 3. Sketch of the ideal extrusion process. The development of the MgSi phases is pointed out in a oversubscribed way

Experiments

The used alloys are in the 6005(A) group. They have a billet diameter of 254 mm and were cast on an industrial scale at our plant in Ranshofen by using Wagstaff equipment. The homogenisation was done with a continuous homogenisation oven from Hertwich. The homogenisation was done at a soaking temperature of 570 °C and a duration of 4h. Afterwards, the billets were cooled by a turbo air cooling system. The cooling rate was set to 500 K/h. The chemical composition of the alloys was determined with the help of an optical spark emission spectroscope SPECTROMAXx [20] and shown in wt% in Table 1. DSC examination was made with the help of a Netzsch DSC204F1. The heating rate was set to 3 K/min.

The samples for all our further experiments were taken out of a serial extrusion process from a very complex side sill. The hourly capacity for this product was approximately 1200 kg/h. The selection is based on the conspicuous low productivity, therefore, following a long dwell time in the gas oven. The gas oven has a length of 7000 mm, and a billet with the diameter of 254mm has 970 kg. Hence, the dwell time of the log was approximately 50 minutes. The settings of the gas oven were: end temperature 435 °C and the previous heating zone gradually colder. The log was heated in the last zone to 435 °C. Directly after reaching the temperature, the slices were cut and quenched. All samples were taken from half the radius of the billet slices. The small R/2 samples had a diameter of 40 mm and a thickness of 25 mm. To simulate the further holding time in the gas oven, the small samples were reheated very quickly in 20 seconds in a salt bath oven. The samples were then kept in the salt bath oven for further 160 seconds, afterwards, the samples were directly water quenched (WQ). The salt bath oven was tempered to 435 °C, 460 °C, 480 °C, 550 °C and 570 °C. The samples were grinded with SiC paper and polished with diamond paste up to 1 µm. The final polishing step was with an oxide polishing suspension from Struers to visualise the MgSi phases. The photos for the analysis were taken with an Olympus BX53 light microscope at a magnification of 1000 times. Smaller particles than 10 pixels were ignored. They were much smaller than the resolution of the light microscope.

The metallographic samples were analysed to get quantitative results of the amount of MgSi and AlFeSi phases. A trained person can conduct the examination of a single picture in approximately 60 minutes. To get a reliable quantitative result you have to analyse 15 pictures (124 x 99 µm) or more in a row if the grain size is approx. 80 µm. Therefore, you do not over or underestimate the uneven distribution of the MgSi and AlFeSi phases depending on the grain boundaries. Consequently, the analysis of the metallographic samples is very time-consuming. To solve the problem an AI was trained to differentiate between MgSi and AlFeSi phases. The AI is capable of analysing 15 pictures in less than 60 seconds.

The AI is based on semantic segmentation, this is a crucial task in machine learning where each pixel in an image is assigned a class. We used a U-Net architecture with a transformer-based approach to pretrain the encoder in an unsupervised way. By leveraging the transformer's sequence processing capabilities, we reduced the required training data. The network was trained using image patches and

an A100 GPU, a powerful hardware component for ML tasks. Training took around 8 hours, varying based on dataset size, model complexity and available resources. Hyperparameter tuning and data augmentation techniques were guided by Wightman et al.'s paper on improved training procedures in time [21]. Our approach yielded a robust semantic segmentation solution.

Table 1. Chemical composition of the used alloy, all contents are given in wt%

	Mg	Si	Fe	Mn	Cr	Al
6005(A)	0.40-0.7	0.50-0.9	max. 0.35	max. 0.50	max. 0.30	res.
6005(A)_1	0.7	0.69	0.2	0.5	0.11	res.

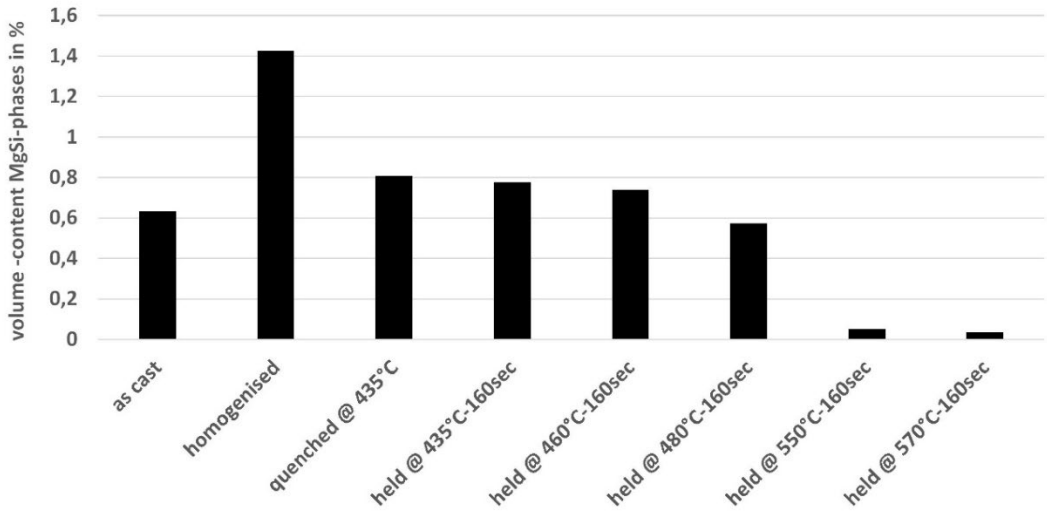
Results

The samples were kept at the mentioned temperatures to simulate the holding time in the gas oven. The normal capacity of an extrusion line is 20 to 30 billets per hour; therefore, the dwell time was set to 3 minutes. The standard production of automotive products also includes the tapering step of the billet in the induction oven to reach an even and stable temperature for the profile directly after the die. This step is very fast compared to all other steps at higher temperatures. The tapering for the billets were 450 °C at the end and 470 °C at the beginning of the billet. This additional time and temperature load was captured with the temperature step of the 480 °C. Higher temperatures were selected to see the influence of a possible overheating of the billet [22,23].

Fig. 4 shows the results of the metallographic analysis of the samples. The as-cast state exhibits that the MgSi phases have 0.6 vol%. The homogenised sample reveals the highest content, it has more than doubled and shows 1.4 vol%. The quenched sample and the sample held for 160 seconds at 435 °C display a decrease of the MgSi volume content. A further increase of the temperature to 460 °C and 480 °C exhibits only another slight decrease of the volume content. The two highest temperatures point out a steep decrease of the MgSi content down to below 0.05 vol%.

The used AI also allows the classification of each MgSi phase. The optical auxiliary conditions gave us the lowest limit of 0.05 µm². The classification steps were 0.1 µm². Fig. 5 exhibits the results of the two main interesting conditions. The homogenised state and the gas oven reheated state. The MgSi phases in the homogenised sample indicate a small size and narrow size distribution. In contrast, the reheated samples via the gas oven have bigger MgSi phases with a much broader distribution. Some further MgSi particles were bigger than 2 µm².

Fig. 6 exhibits the DSC measurement of the examined alloy. The first visible precipitation peak for MgSi phases is located approximately at 264 °C. The second peak is the dissolution peak placed at 400 °C, followed by the next precipitation peak at around 445 °C. At the end of the DSC sequence there is the dissolution peak.



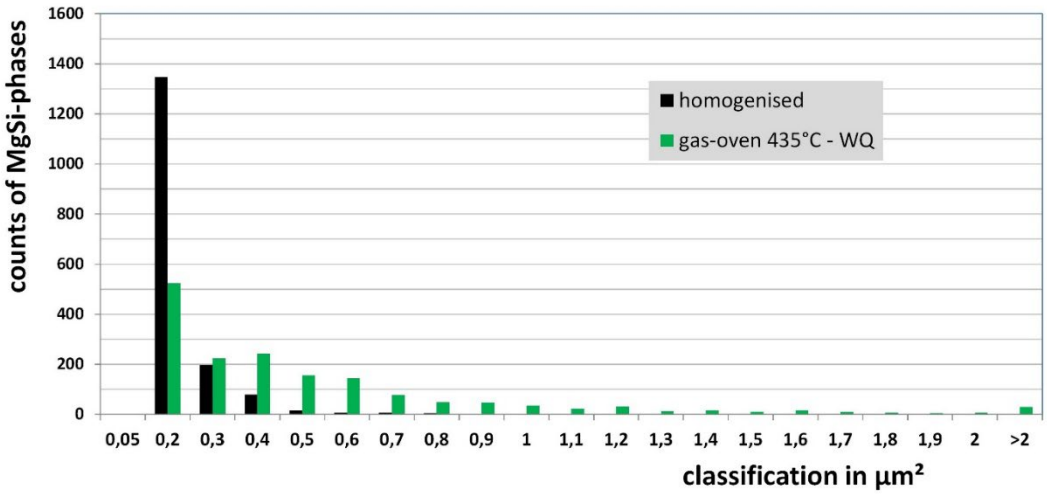


Fig. 4. Results for the counts for each classification slot for the MgSi phases

Discussion

From the point of view of the extrusion process, it does not matter if the MgSi phases in the reheated billet were β, β` or β``phases. They all should dissolve during the extrusion process to reach the solute saturated state (SSS). Afterwards, this SSS can be quenched in to get the highest mechanical properties after the heat treatment or to prevent some surface issues caused by the remelting of big MgSi phases. The extrusion parameter was set to a gas oven temperature of 435 °C. This gas oven temperature was formerly considered a good compromise between the mechanical properties and the possibility to hot shear the billet. The hot shearing process was mandatory before the use of the hot saw to cut the log into the desired billet length for the extrusion process. From the literature it is well known that the β or β` phases prefer to precipitate at the temperature range from 360 °C up to 380 °C [24–26].

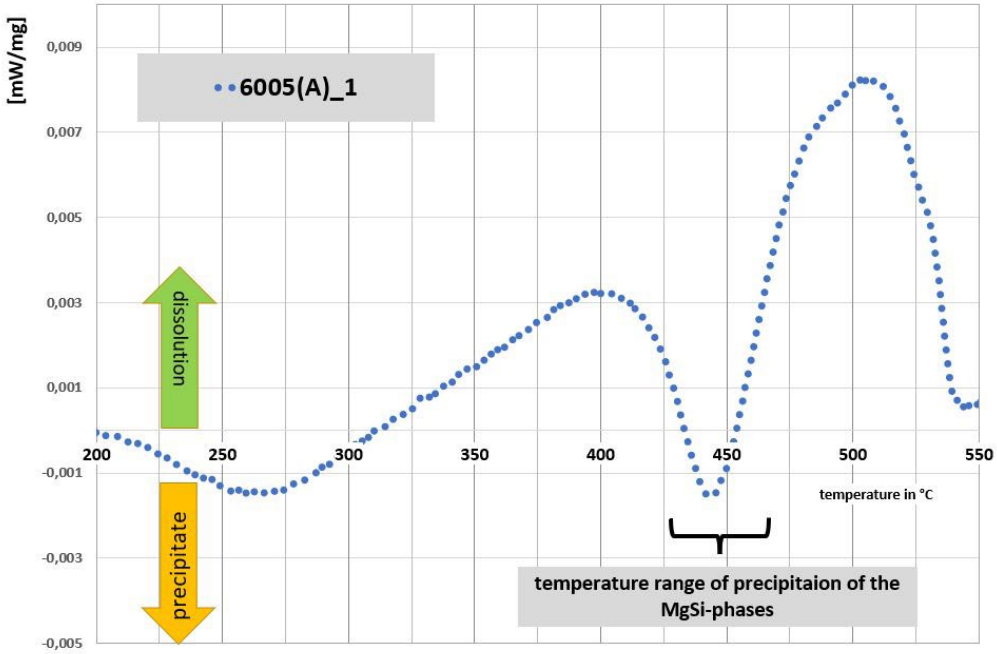


Fig. 5. DSC measurement of the used 6005(A) alloy. The curve presents the temperature range were the MgSi phases should precipitate or dissolve

The tested 6005(A) alloy exhibits a shift to a higher temperature range up to 445 °C. This effect could be influenced by the used initial state. Normally, the alloys were tested in the solutionised state, therefore, the driving force to precipitate is higher and the phases precipitate at lower temperatures.

The tested sample was in the as-cast state, therefore, the major percentage of the MgSi phases was captured in the big primary MgSi phases. Hence, the favoured precipitation temperature was higher.

The DSC results exhibit that exactly in that temperature range of 445 °C +/- 10° the precipitation of the MgSi phases has its maximum. From this perspective, the used gas oven temperature of 435 °C is not the perfect match. The MgSi phases have the best conditions to precipitate and to grow. This can be seen in Fig. 4 and Fig. 5, the MgSi phases were not dissolved. The smaller MgSi phases from the homogenised state were dissolved and were consumed from the bigger ones supported from the Ostwald ripening process. These bigger phases need, in the further processing steps, more time or more deformation at higher temperatures to dissolve. The MgSi volume content is very stable in the range of 435 °C up to 460 °C. The slight temperature increase to 480 °C indicates a small reduction of the volume content. The exposure time at 480 °C and 160s was too short to dissolve the bigger particles, only the small ones were gone. But with the used billet quantity per hour, the holding time cannot increase further. The only way to reduce the MgSi content was to raise the temperature up to 550° and 570 °C. Only some big MgSi phases survive at this high temperature at an exposure time of 160s. But the residual volume content is very low compared to all other conditions, < 0.05 vol%. A temperature rise to 460 °C or higher is not possible in the industrial extrusion process. Additional heat arises during the deformation of the billet. This additional temperature plus the temperature of the reheated billet is too high for a good extrusion process. If all other parameters of the extrusion process are held constant. This leads to transverse tears of the profile melting of the grain boundaries [27].

The 550 °C and higher were only a test to see a correlation between time and temperature and the dissolution of MgSi phases. The 570 °C was the same temperature as to homogenisation temperature. The exposure time of 160 seconds was enough for both temperatures to dissolve the MgSi phases which precipitate during the cooling after the homogenisation heat treatment and the coarsened MgSi phases during reheating of the billet. This is good to know. If a taper quench is installed, the overheating and quenching of the billet can solve the problem of the undissolved MgSi phases and enhance the extrusion process [28].

Conclusion

The experiments with the gas oven were done in industrial scale to get the perfect samples as starting point for the heat treatment experiments. Afterwards the samples were treated in a salt bath oven to simulate different temperatures in the gas oven at a constant duration. This chosen experimental setup is very close to the industrially possible processes.

The use of a trained AI has proven to be useful to analyse the MgSi phases. The time to label and count the different phases was decreased significantly. At the same time, the number of analysed pictures can be increased to get reliable results.

The historical approach of using the same parameters at the gas oven with new equipment has proven to be false. The parameters used in the past were set to this high temperature to shear the log. Using a hot saw and keeping the DSC results of the used alloy in mind, the temperature range was set perfectly into the precipitation maximum of the MgSi phases. The homogenised sample shows the highest volume content of MgSi phases but the smallest ones. Reheating of the billet makes the smallest MgSi phases dissolve, but the bigger ones grow of the expanse of them. A further increase of the billet temperature was not useful. The raised temperature leads to tearing of the profile.

On the one hand considering the DSC measurements, the temperature could be decreased down to 380 °C - 400 °C to prevent the excessive precipitation of the MgSi phases. On the other hand, the use of a taper quench at temperature higher than 550°C can be useful to enhance the extrudability in perspective of reducing the volume content of undissolved MgSi phases in the billet.

Acknowledgement

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