Optimizing Heat Treatment Parameters for the W-Temper Forming of 7xxx Series Aluminum Alloys

Shreyas Hebbar, Lukas Kertsch * and Alexander Butz

Fraunhofer Institute for Mechanics of Materials IWM, Wählerstr. 11, 79108 Freiburg, Germany; hebbarshreyas13@gmail.com (S.H.); alexander.butz@iwm.fraunhofer.de (A.B.)
* Correspondence: lukas.kertsch@iwm.fraunhofer.de; Tel.: +49-761-5142-479

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Abstract: A major challenge in processing 7xxx series aluminum alloys is their limited formability at room temperature. In this paper, for the alloys EN AW-7020 and EN AW-7075, various variants of the W-temper forming process are investigated. For both alloys, a good cold formability and a high strength after aging can be achieved. The effects of solution heat treatment or retrogression temperature and holding time, as well as the influence of plastic deformation after quenching, were studied. For various combinations of process parameters, the formability of the as-quenched materials and the hardening performance during artificial aging were examined. For this, hardness measurements and differential scanning calorimetry (DSC) experiments were performed along the entire process chain, to reveal the development of the hardening precipitates. After solution heat treatment and quenching, the yield stress and hardness of both investigated alloys were drastically reduced in comparison to their initial T6 states, while the ductility was significantly increased. By a subsequent simple artificial aging treatment, the same hardness as in the T6 state could be restored. It was observed that plastic deformation immediately after quenching significantly decreased the artificial aging time to achieve the peak hardness. Besides the conventional solution heat treatment process, an alternative retrogression and re-aging procedure was identified for the alloy EN AW-7020. While the heat treatment temperature can be reduced as compared to the conventional solution heat treatment, the formability and hardenability are equally good. In contrast, no such alternative process could be identified for the alloy EN AW-7075.

Keywords: 7xxx series aluminum alloys; W-temper forming; room temperature forming; solution heat treatment; retrogression and re-aging; precipitation

1. Introduction

Due to the high strength-to-weight ratio of 7xxx series aluminum alloys, they gain increasing attention for producing high performance sheet metal components. These heat treatable alloys owe their strength to the formation of precipitates during heat treatment processes [1]. The main alloying elements of this series are zinc and magnesium, and sometimes copper, providing the highest strengthening potential among all age-hardenable aluminum alloys. Therefore, alloys of this series have great potential for application as high strength sheet materials in the aerospace and automotive industry [2]. In order to achieve the desired strength of the final product, in industrial applications, the material is heated above the temperature at which the strengthening precipitates dissolve and subsequently quenched to a supersaturated solid solution. While, by a solution heat treatment at high temperatures the hardening precipitates dissolve completely, in retrogression at moderate temperatures the dissolution is usually incomplete. A critical quenching rate has to be maintained to avoid undesired precipitation during cooling [3]. From the supersaturated solid solution, the precipitation process takes place in three successive stages: nucleation, growth, and coarsening. During nucleation, spontaneously
forming clusters of solutes turn into small particles that are able to grow. By diffusion of further solutes from the solid solution to the existing precipitate nuclei, they grow until close to the maximum achievable volume fraction. If this process happens at room temperature, the process is called natural aging, whereas it is called artificial aging if it is done at elevated temperatures. During the growth stage, the strength of the material increases significantly. By further aging, coarsening leads to an increase of precipitate size and a decrease of particle density at an almost constant volume fraction, leading to a loss of strength. Furthermore, the existing precipitates can transform into other phases of higher thermodynamic stability. Excessive coarsening and the formation of phases that are less effective for strengthening is called over-aging and is usually not desired. The sequence of these steps in the decomposition process depends on the proportions of the alloying elements, Mg/Zn ratio [4], heat treatment temperature [5,6], cooling rate [7,8] and aging temperatures [3,9,10]. There are various works on the evolution of precipitates in 7xxx series aluminum alloys [11–15], and a recent review is given by [4]. The general precipitation sequence can be summarized [10,13] as

\[
\text{Supersaturated solid solution} \rightarrow \text{GP zones} \rightarrow \text{metastable } \eta' \rightarrow \text{stable } \eta.
\]

Guinier-Preston (GP) zones are solute-rich regions forming during room temperature aging and early stages of artificial aging. These precipitates act as precursors to the metastable \( \eta' \) phase. The metastable \( \eta' \) phase is recognized as the main hardening phase contributing to the peak hardness in Al-Zn-Mg(-Cu) alloys [13]. At later stages of artificial aging, the metastable \( \eta' \) precipitates may serve as nuclei for stable precipitate phases. The most common stable phase is \( \eta (\text{MgZn}_2) \), but there are other stable phases, such as the T phase (\( \text{Mg}_{32}(\text{Al,Zn})_{49} \)) and S phase (\( \text{Al}_2\text{MgCu} \)) [4], which may occur during slow cooling. The formation of these stable phases leads to a decrease in strength [16]. Besides a favorable composition, optimized casting conditions may significantly increase the strength [15].

The addition of Cu to Al-Zn-Mg alloys increase the achievable strength and reduce the time required for aging. Due to a higher supersaturation, it increases the solid solution strengthening and reduces the nucleation radius of precipitates, leading to faster precipitation and allowing a higher precipitate volume fraction before transforming to more stable precipitates [17]. Furthermore, Cu-rich vacancy-solute clusters form early after quenching assisting the formation of \( \eta' \) [18]. While decreasing the susceptibility to the rate of heating to the aging temperature, the addition of Cu increases the quench sensitivity [17]. Furthermore, Cu enhances the corrosion resistance [19].

One major challenge in the manufacturing of these alloys is to achieve a sufficient formability for the production of sheet and tube components and to ensure high strength in service. Cold forming is a beneficial process, but it only allows a limited freedom of design due to the poor ductility of the aged material, which is not beneficial for producing complex-shaped parts. On the contrary, warm forming requires great effort since the blank and the forming tool must be heated, conditioned, and maintained. To increase the formability of hardenable high-strength alloys at room temperature, a temperature-assisted process named W-temper forming can be utilized [20]. As Figure 1 shows, in this process, the sheet material is heat treated, quenched to room temperature and formed within a short time after quenching. Subsequently, the formed parts are aged to regain the maximum strength. The W-temper process enhances the potential for inline series forming operations of 7xxx series aluminum alloys for industrial applications. In addition, the process allows a relatively complex tool design and the application of a wide range of lubricants.

The goal of the current work is to provide quantitative information about the influence of the W-temper forming process parameters on the formability and final material properties, such as the achievable hardness and microstructure. Special focus will be put on an efficient process design, which is applicable to industrial use cases of 7xxx series aluminum alloys. The goal of the current work is to provide quantitative information about the influence of the W-temper forming process parameters on the formability and final material properties, such as the achievable hardness and microstructure. Special focus will be put on an efficient process design, which is applicable to industrial use cases of 7xxx series aluminum alloys.
2. Materials and Methods

2.1. Materials

In this work, the two commercial aluminum alloys EN AW-7020 and EN AW-7075 are investigated. They were received as sheets of 2 mm thickness in a T6 temper state. The chemical compositions of both alloys are given in Table 1. The sheets were further sectioned into tensile specimens with a gauge length of 65 mm and a width of 10 mm. The tensile specimens were cut along the rolling direction.

| Alloy      | Al   | Zn   | Mg   | Cu  | Fe  | Si  | Mn  | Cr  | Zr  | Ti  |
|------------|------|------|------|-----|-----|-----|-----|-----|-----|-----|
| EN AW-7020 | 93.14| 4.92 | 1.27 | 0.06| 0.13| 0.05| 0.15| 0.10| 0.09| 0.02|
| EN AW-7075 | 87.54| 7.26 | 2.92 | 1.72| 0.11| 0.08| 0.03| 0.19| 0.04| 0.04|

2.2. Heat Treatment

The heat treatments were performed in the thermo-mechanical simulator Gleeble 3150 (Dynamic Systems Inc., Poestenkill, NY, USA) using direct resistance heating to heat the samples. Figure 2a shows the Gleeble test setup. The samples are placed between the two steel jaws, which are internally water-cooled. The specimen temperature during heating and cooling is monitored and controlled by a type K thermocouple welded at the center of the sample. The maximum temperature is reached in the middle of the sample, whereas the temperature decreases with increasing distance from the center, as heat is conducted out through cold steel jaws. Hence, all material characterizations (hardness measurements, differential scanning calorimetry (DSC) measurements, and optical microscopy) were done within a distance of less than 8 mm from the center of the heat-treated samples, which was selected as a compromise between having a sufficient area for material characterization and minimizing the deviation of the temperature from its maximum value in the center of the specimen. Tests were repeated twice to ensure reproducibility.

Based on temperature, two types of heat treatments can be distinguished: in the solution heat treatment (SHT) process, the alloy is heated above the solvus temperature of the hardening precipitates and held at that temperature to obtain a homogeneous solution [21]. In the second case, the alloy is heat-treated for a short period of time at a temperature between the age-hardening temperature and the SHT temperature. This process is called retrogression [22].

A heating rate of 20 K/s was selected for performing all heat treatments. Quenching after the heat treatment was realized with compressed air from a pair of nozzles pointing at the center of the samples. The temperature decrease during air quenching in the Gleeble is shown in Figure 2b. Average cooling rates of 147 K/s between 425 °C and 180 °C, as well as 126 K/s between 320 °C and 180 °C, were maintained. This is faster than the critical rates to avoid the quench-induced precipitation reported in literature. The critical quenching rate to suppress precipitation during quenching is 3 K/s for the alloy.
EN AW-7020 [7] and 100 K/s for the alloy EN AW-7075 [8]. To compare the outcome of the various thermo-mechanical processes performed in this work, all specimens were artificially aged in a furnace at 120 °C. It was not aimed at the simultaneous optimization of the aging procedure.

In this work, DSC was used to analyze the phase formation processes. In fact, this technique only provides indirect insights and does not offer any information about the size and location of precipitates. However, DSC was chosen, because it is fast and very sensitive, such that even small changes of the phase structure can be detected. More information about the types of precipitates as well as about their location and size can be gained by transmission electron microscopy (TEM). While DSC measurements show the effective behavior of samples of a finite size, TEM only offers insights into a very small region.

In order to conclude about the effective material behavior, many different measurements are required. This makes DSC a superior technique for a fast screening of the effect of various process variants.

DSC measurements of both alloys were performed in the as-received condition and after various thermo-mechanical processes using a Pegasus 404C (Netzsch Gerätebau GmbH, Selb, Germany). The samples were heated to 500 °C with a heating rate of 10 K/min. The endothermic and exothermic peaks in the resulting plots of temperature versus heat flow correspond to the dissolution and formation of precipitates, respectively.
2.5. Optical Microscopy

The as-received material and different heat-treated samples were characterized by metallographic methods. After mounting, grinding and polishing, the samples were etched to reveal their microstructures. For the alloy EN AW-7020, the Murakami etchant (5 g K$_3$(Fe(CN)$_6$), 10 g NaOH, and 60 mL distilled water), and for the alloy EN AW-7075, a 20% NaOH etchant were used. The microstructures were then analyzed in a Nikon Eclipse ME600 optical microscope (Nikon Corporation, Chiyoda, Tokyo, Japan). The average grain size was determined with the linear intercept method.

3. Results and Discussion

The strategy to gain the maximum insight into the behavior of both considered materials was to first investigate the alloy EN AW-7020, because its chemical composition is less complex. After having understood the key features of its behavior, further investigations were made for EN AW-7075, but to a lesser amount. This alloy contains more Zn and Mg and furthermore it bears a considerable amount of Cu. However, it forms similar hardening phases [17], such that its qualitative properties and behavior are expected to be quite similar to those of EN AW-7020. In addition, performing experiments with the higher alloyed material was significantly more difficult, because welding the thermocouples is more challenging and they often detach at high temperatures.

3.1. Heat Treatment Temperatures

At first, appropriate heat treatment temperatures were defined with the aid of DSC measurements. The DSC heating curves provide information about the temperatures, at which certain precipitates dissolve and form. DSC heating experiments were done with both alloys, starting with their as-received states. The resulting heat flow curves are shown in Figure 3. In this representation, the upward peaks indicate endothermic reactions caused by the dissolution of precipitates, whereas downward peaks indicate exothermic reactions due to the formation of new precipitates. The results were analyzed based on the findings in [6,23].

![Figure 3. Differential scanning calorimetry (DSC) heating curves for the as-received alloys, (a) EN AW-7020 and (b) EN AW-7075, at a heating rate of 10 K/min.](image)

In Figure 3a, for the alloy EN AW-7020, the first endothermic peak A can be related to the dissolution of the metastable $\eta'$ precipitates [6]. The following exothermic peaks B and C indicate the formation of stable $\eta$ precipitates, possibly overlapping with the later stage of dissolution of coarse $\eta'$ particles [10]. In the temperature interval of 250–350 °C, an exothermic peak D can be observed, which can be ascribed to progressive dissolution of stable $\eta$ precipitates [24]. Similar interpretations...
were made to observe the dissolution behavior for the alloy EN AW-7075, as shown in Figure 3b. Again, the first endothermic peak A at 190 °C is related to the dissolution of metastable η' precipitates. The exothermic peak B at the temperature of 220 °C relates to the formation of the stable η phase. Above this temperature, the dissolution of η precipitates can be observed. The peak C might be due to the coarsening of some of the η precipitates overlapped with the onset of dissolution of other η precipitates [24]. On further heating, it is observed that the stable Mg-Zn phases completely dissolve up to a temperature of 435 °C. A sharp exothermic peak E at approximately 490 °C probably relates to the dissolution of the β (Mg$_2$Si) phase [8].

From the determined DSC curves, suitable heat treatment temperatures can be determined. For both alloys, 480 °C was chosen as the solution heat treatment (SHT) temperature. Based on the dissolution of the metastable η' and stable η precipitates which contribute most to the hardness of the material, different retrogression temperatures were classified for both alloys. At first, for the alloy EN AW-7020, a lower retrogression temperature of 225 °C (assuming complete dissolution of η' precipitates), a higher retrogression temperature of 360 °C (completion of η precipitates dissolution) and an intermediate temperature of 330 °C were chosen. After having evaluated the results of the prior alloy, for the alloy EN AW-7075, only the higher retrogression temperatures of 320 °C, 350 °C and 380 °C were further investigated. The lower retrogression temperature, however, only led to poor results, as will be demonstrated in the following section.

### 3.2. Holding Time for the Alloy EN AW-7020

The minimum holding time required for different treatment temperatures was initially investigated for the alloy EN AW-7020. For this, the mechanical properties were determined immediately after various holding times at the solution heat treatment temperature of 480 °C and different retrogression temperatures. Hardness measurements (Figure 4a) and tensile tests (Figure 4b,c) were performed after heat treatments at various holding times, followed by air quenching. At 480 °C, a holding time of 5 s was sufficient to reduce the hardness from 121 HV0.5 to 51 HV0.5. Further increasing the holding time did not significantly change the hardness after SHT and quenching. Figure 4c shows the tensile curves after SHT with two different holding times, leading to a very similar flow behavior. The serrations of the stress-strain curves can be attributed to the Portevin–Le Chatelier effect (PLC effect) [25].

The time to reduce the hardness during retrogression treatments strongly depends on the retrogression temperatures (see Figure 4a). For the lower retrogression temperature of 225 °C, the hardness decreased as the holding time was increased. However, even after a time of 120 s, the reduction in hardness was not significant as compared to the as-received T6 state. It can be supposed that, with increasing the holding time, the η' precipitates transform to stable η precipitates [6].

Due to the industrial demand of short process times, times beyond 120 s were not examined. The higher retrogression temperatures of 330 °C and 360 °C led to similar hardness values as in the SHT process. From the stress-strain curves recorded after retrogression after different temperatures and holding times (Figure 4b), a substantial decrease in stress and increase in total elongation in comparison to the initial T6 state can be observed as the retrogression temperature increased. Similar findings were reported by Unger [26] for a similar alloy. The point of failure and the ultimate tensile strength for the higher retrogression temperatures and SHT could not be measured, because the samples reproducibly broke outside the gauge length. With a retrogression temperature of 225 °C, increasing the holding time from 5 s to 60 s led to a reduction in yield strength. However, the ductility was poor even after the longer holding time, resulting in a poor formability. Using the higher retrogression temperature of 360 °C, the ductility increased to a similar level as after SHT. The PLC effect after retrogression at 360 °C was slightly weaker than after the SHT process, while it was absent after retrogression at 225 °C. Finally, a holding time of 30 s was chosen for both SHT and retrogression, to allow the solutes to homogeneously distribute within the material.
Figure 4. Change of mechanical properties of the alloy EN AW-7020 after various thermo-mechanical processes. (a) As-quenched hardness values after heat treatment processes at various holding times for different temperatures. (b) Room temperature stress-strain curves at a strain rate of 0.1 s$^{-1}$ after various heat treatment processes. (c) Room temperature stress-strain curves at a strain rate of 0.01 s$^{-1}$ after solution heat treatment (SHT) (480 °C) with two different holding times.

3.3. Aging Response and Effect of Pre-Deformation on the Alloy EN AW-7020

The age hardening process for the alloy EN AW-7020 due to the evolution of precipitates was investigated by Vickers hardness measurements. Figure 5 shows the evolution of hardness over the aging time. On the one hand, specimens subjected to SHT were artificially aged directly after quenching. On the other hand, in order to identify the influence of a plastic pre-deformation on the artificial aging process, some samples were strained in the Gleeble by 15%, at a strain rate of 0.1 s$^{-1}$, immediately after SHT and air quenching.

The aging performance of these samples is shown in Figure 5a. The hardness prior to artificial aging without pre-deformation was 51 HV0.5 in contrast to 75 HV0.5, with pre-deformation due to strain hardening. In the initial stages of artificial aging, the hardness increased, due to the formation of GP zones [11]. With increasing aging time, GP zones grow and transform into metastable η’ precipitates [11]. For the samples without pre-deformation, most of the precipitates are assumed to be homogeneously distributed, except for precipitate free zones close to grain boundaries. On the contrary, pre-straining the samples introduces a high dislocation density [10], leading to heterogeneous precipitation. The time to peak hardness in the pre-deformed samples as compared to the undeformed material, which is ascribed to an increased number of nucleation sites and an enhanced diffusivity, leading to faster precipitation [27]. Peak hardness values of 121 HV0.5 after 42 h and 118 HV0.5 after
27 h were achieved for the undeformed and pre-strained samples, respectively. This means that in the undeformed material, exactly the as-received hardness could be restored. Although the strain hardening may not be completely recovered when the peak hardness is reached, the peak hardness with pre-deformation was 2.5% lower than without deformation. This might indicate a coarser precipitate structure, due to the presence of dislocations [10]. After having reached its peak value, the hardness started decreasing, due to over-aging. The decrease in hardness after peak aging was faster in the pre-strained samples. The reason for this is probably that, on the one hand, the precipitates are already coarser when the peak hardness is reached, and on the other hand, the coarsening kinetics is accelerated if a certain amount of dislocations have not yet recovered at this time [10].

![Figure 5](image)

**Figure 5.** Evolution of the hardness during artificial aging of the alloy EN AW-7020 after various thermo-mechanical processes. (a) Effect of pre-deformation after SHT (at 480 °C) or retrogression at 360 °C, with a holding time of 30 s. (b) Effect of holding time during retrogression at 225 °C. (c) Effect of temperature during retrogression with a holding time of 30 s.

Figure 5b demonstrates the effect of artificial aging for different retrogression holding times at 225 °C. For holding times up to 30 s, the very high as-quenched hardness indicates a poor formability, and even after an aging time of 42 h, the initial hardness could not be restored. After holding times of more than 30 s, the hardness was significantly reduced (93 HV0.5 after 60 s and 90.8 HV0.5 after 120 s). However, after 24 h of aging, the hardness did not further increase and remained at intolerably low values. Aging for more than 42 h was not considered, because this would not be an option for industrial applications. Altogether, it is obvious that retrogression at 225 °C is not feasible.

Figure 5c illustrates the aging behavior for specimens subjected to retrogression heat treatments of 30 s duration at different temperatures. Both 330 °C and 360 °C were sufficient to decrease the
as-quenched hardness to a similarly low value as after SHT. During aging, however, the hardness of the samples heat-treated at the higher of the two retrogression temperatures increased faster. Combining this observation with the aging performance shown in Figure 5a makes clear that retrogression at a temperature of 360 °C is a promising alternative process to SHT. With 15% pre-deformation after the retrogression treatment, a peak hardness of 119 HV0.5 was achieved, which is only 1.7% less than the as-received hardness. Without pre-deformation, the peak hardness was 114 HV0.5 (5.8% less than as-received).

In order to verify that the various heat treatment processes result in the assumed dissolution and precipitation reactions described above, further DSC measurements were performed. Figure 6a shows the DSC curve immediately after SHT and quenching. The absence of endothermic peaks below 250 °C indicates that the η′ precipitates were dissolved as desired. Figure 6b shows the curves after SHT and aging for different times. After 24 h (peak aged), the curve is very similar to that of the as-received material (see Figure 3a). The peaks B, C, and D of the corresponding solid blue curve are related to the dissolution of η′, the formation of η, and the dissolution of η precipitates, respectively. The distinct peak B indicates that, actually, a high content of the hardening η′ phase was present in the peak-aged state. In comparison, after only 6 h (under-aged, dash-dotted red line), the peak 1B is very weak, indicating that there was only a little amount of the η′ phase. On the other hand, the endothermic peak 1A shows that a certain amount of GP zones were present, what is typical for an early state of precipitation. Finally, after 62 h (over-aged, dashed green line), a quite similar behavior as in the peak-aged state was observed. However, the intensity of peak B is relatively lower and that of peak D is higher, while peak C, indicating the formation of η, did not significantly change. This shows that after long aging times, there is a lower content of metastable η′ precipitates and already a higher content of the stable η phase [28]. In addition, Figure 6b displays the DSC curve of a sample after retrogression and re-aging (RRA) for 42 h (dotted magenta line), which is very similar to the curve after SHT and peak aging. However, the intensity of peak B indicating η′ dissolution is slightly lower in the RRA curve. Therefore, a slightly lower η′ content after the retrogression procedure might be the reason for the somewhat lower attainable peak hardness as compared to the SHT process (see Figure 5a).

![Figure 6](image1)

**Figure 6.** DSC curves for the alloy EN AW-7020 performed with a heating rate of 10 K/min. (a) Immediately after SHT and quenching, (b) at intermediate stages of artificial aging after solution heat treatment (SHT) or retrogression and re-aging (RRA).

Although it seems that the curves of the specimens after SHT and aging for 24 and 62 h as well as after RRA show an exothermic peak around 150 °C, this is probably not a distinct reaction. In fact, η′ may form in this temperature range. If the peak was related to this reaction, it should be strongest immediately after SHT (Figure 6a) and weakest after SHT and aging for 42 h or more (Figure 6b, solid blue and dashed green). However, since the opposite trend is observed, the apparent exothermic peak
Besides the maximum attainable strength, pre-deforming the material might affect its thermal stability. On the one hand, if any strain hardening remains after the aging treatment, recovery under service conditions at elevated temperatures or even at room temperature might reduce the strength. However, this effect should not be very pronounced after the relatively long ageing times used here. On the other hand, precipitate coarsening under service conditions can strongly deteriorate the strength [29]. As discussed above, there is an indication that the precipitates are coarser if a forming operation is involved. Therefore, the coarsening might be faster or set in earlier than in an undeformed material. This issue has to be considered in the process design.

### 3.4. Aging Response and Effect of Pre-Deformation on the Alloy EN AW-7075

Similar investigations were made for the alloy EN AW-7075. The stress-strain curves for the as-received and solution heat treated samples are shown in Figure 7a. Solution heat treatment led to a significant reduction in yield strength to approximately one third of the strength of the as-received material. However, after SHT, the samples reproducibly fractured at a strain of around 8%, due to necking outside the gauge length (see Figure 7b). Because the applied method could not provide sufficient information of the ductility, we refer to a similar investigation in reference [5]. From this investigation, it turns out that the ultimate yield strength after a heat treatment at 300 °C is lower than after SHT at 480 °C. This led to early failure in that region of the specimen where the temperature did not exceed 300 °C. The stress-strain curves reported in [5] are in very good agreement with the ones presented here. This reference suggests that after SHT at 480 °C, a maximum elongation of more than 20% can be expected. At strains higher than 4%, the PLC effect caused some serrations in the stress-strain curve.

![Stress-strain curves of EN AW-7075 before and after SHT](image1)

**Figure 7.** (a) Stress-strain curves of EN AW-7075 before and after SHT. (b) Specimen of a tensile test after SHT which, as in all such tests, broke outside the gauge length; the red arrow points at the center with reference to the undeformed specimen.

Because such early failure was observed in the tensile tests, for this alloy, a pre-strain of only 7% was used to further study the effect of pre-deformation on the aging. At higher strains, there is the risk of undesired failure during the pre-deformation. Prior to artificial aging, hardness values of 94 HV0.5 and 120 HV0.5 were recorded without and with pre-deformation, respectively. Figure 8a shows the evolution of hardness during artificial aging after solution heat treatment. It was possible to restore the as-received T6 hardness of 190 HV0.5 for all samples, without and with pre-deformation. The time to peak hardness is 24 h without pre-deformation and it is reduced to 15 h by pre-straining the material, indicating faster precipitation due to an increased number of nucleation sites and an enhanced diffusivity [27].
weak peak 2A indicating that only a low volume fraction of the hardening phase \( \eta \) to those of the as-received T6 material.

The peak aged sample in Figure 9a. This confirms that the precipitates formed during aging are similar line) and 24 h aging (peak aged, solid red line). The curve of the under-aged material shows a very curve in Figure 3b, illustrating that the Mg-Zn phases are completely dissolved above 435 °C of the peak aged sample in Figure 9a. This confirms that the precipitates formed during aging are similar to those of the as-received T6 material.

However, the achievable peak hardness was considerably less than after SHT. We suggest that this is due to an incomplete dissolution of the existing hardening precipitates during the retrogression heat treatments. Hence, during aging, the existing precipitates can coarsen or transform into stable precipitates, which are less effective for hardening. This observation can be directly related to the DSC curve in Figure 3b, illustrating that the Mg-Zn phases are completely dissolved above 435 °C (peak D). Consequently, no alternative heat treatment temperature was identified for this alloy, which would both sufficiently increase the cold formability and provide a satisfying aging response.

Figure 9 displays the DSC results of specimens after heat treatment at various temperatures and artificial aging. Figure 9a shows the measurements after SHT and 3 h aging (under-aged, dashed blue line) and 24 h aging (peak aged, solid red line). The curve of the under-aged material shows a very weak peak 2A indicating that only a low volume fraction of the hardening phase \( \eta' \) has precipitated during the first 3 h of aging. The first distinct peak 2B can be linked to the formation of stable \( \eta \) precipitates [28]. The curve of the as-received material (Figure 3b) shows the same peaks as the one of the peak aged sample in Figure 9a. This confirms that the precipitates formed during aging are similar to those of the as-received T6 material.

The lowest retrogression temperature of 225 °C was not investigated for the alloy EN AW-7075, because the analysis of EN AW-7020 showed that this temperature results in an insufficient cold formability. Figure 8b displays the hardness evolution during artificial aging after retrogression at three intermediate temperatures. The as-quenched hardness prior to aging was similar as after SHT. However, the achievable peak hardness was considerably less than after SHT. We suggest that this is due to an incomplete dissolution of the existing hardening precipitates during the retrogression heat treatments. Hence, during aging, the existing precipitates can coarsen or transform into stable precipitates, which are less effective for hardening. This observation can be directly related to the DSC curve in Figure 3b, illustrating that the Mg-Zn phases are completely dissolved above 435 °C (peak D).

Figure 9 shows the DSC results of specimens after retrogression at various temperatures and aging for 24 h. After retrogression without subsequent deformation, (b) retrogression without subsequent deformation. The lowest retrogression temperature of 225 °C was not investigated for the alloy EN AW-7075, because the analysis of EN AW-7020 showed that this temperature results in an insufficient cold formability. Figure 8b displays the hardness evolution during artificial aging after retrogression at three intermediate temperatures. The as-quenched hardness prior to aging was similar as after SHT. However, the achievable peak hardness was considerably less than after SHT. We suggest that this is due to an incomplete dissolution of the existing hardening precipitates during the retrogression heat treatments. Hence, during aging, the existing precipitates can coarsen or transform into stable precipitates, which are less effective for hardening. This observation can be directly related to the DSC curve in Figure 3b, illustrating that the Mg-Zn phases are completely dissolved above 435 °C (peak D). Consequently, no alternative heat treatment temperature was identified for this alloy, which would both sufficiently increase the cold formability and provide a satisfying aging response.

Figure 9 displays the DSC results of specimens after heat treatment at various temperatures and artificial aging. Figure 9a shows the measurements after SHT and 3 h aging (under-aged, dashed blue line) and 24 h aging (peak aged, solid red line). The curve of the under-aged material shows a very weak peak 2A indicating that only a low volume fraction of the hardening phase \( \eta' \) has precipitated during the first 3 h of aging. The first distinct peak 2B can be linked to the formation of stable \( \eta \) precipitates [28]. The curve of the as-received material (Figure 3b) shows the same peaks as the one of the peak aged sample in Figure 9a. This confirms that the precipitates formed during aging are similar to those of the as-received T6 material.

Figure 9. DSC curves for EN AW-7075 at a heating rate of 10 K/min. (a) After SHT and aging for 3 h and 24 h, (b) after retrogression at various temperatures and aging for 24 h.
Figure 9b shows the DSC results of specimens after retrogression at three different temperatures and 24 h artificial aging. The curve for the lowest retrogression temperature of 330 °C does not show any recognizable peak at low temperatures, and only a weak peak 1B, indicating the formation of the η phase. This means that during the retrogression heat treatment, most of the η’ precipitates must have transformed to stable precipitates. For the next higher retrogression temperature of 350 °C, the picture is similar. There, however, the peak 2B is stronger, such that we can conclude that a lower volume fraction of the η phase has formed at this temperature. Nevertheless, the absence of endothermic peaks A around 200 °C indicates that, after both retrogression heat treatments, a negligible amount of the hardening η’ phase is precipitated. In contrast, for the retrogression at 380 °C, there is a considerable peak 3A, demonstrating the presence of a significant amount of η’ precipitates after aging. However, both the endothermic peak 3A and the exothermic peak 3B are still weaker than the corresponding peaks in Figure 9a (SHT and peak-aged) and Figure 3b (as-received T6). This means that even the highest investigated retrogression temperature leads to the formation of a certain amount of the η phase reducing the potential for the precipitation of η’ during aging. This explains why the observed hardness after aging is not satisfying (see Figure 8b).

For this alloy, too, the question of thermal stability arises. At least from the results shown in Figure 8a, there is no indication that precipitate coarsening is more pronounced in the pre-deformed material. The peak hardness without and with pre-deformation is the same, and the rate of hardness drop during overaging is very similar. However, due to the limited investigation of the overaging regime, no definite conclusion can be drawn at this point.

3.5. Optical Micrographs

The as-received materials exhibit a grain structure, which is compressed perpendicular to the rolling direction. For the case of EN AW-7020, this can be seen in the micrographs with view along the sheet normal direction (ND) and transverse direction (TD), see Figure 10a,b. Table 2 summarizes the results of the grain size measurements. The average grain size of the as-received sheet is 45 µm. None of the various heat treatments induced a significant change of the grain morphology and grain size. Even including a deformation after SHT (Figure 10c,d) did not lead to considerable changes. After solution heat treatment, the average grain size slightly increased to 56 µm. The reason for this may be that, at the high temperature of 480 °C, in the absence of pinning precipitates, some grain growth is possible. The aging, however, did not further change the grain size. Apparently, the low aging temperature of 120 °C and the developing precipitates inhibit any grain growth.

![Figure 10. Cont.](image-url)
was developed. The effectivity of the analyzed process variants was evaluated in terms of the softening and ductility increase after the heat treatments, as well as the hardening potential during artificial aging. The hardening potential was determined from a simplified one-step artificial aging treatment.

For both alloys, after solution heat treatment, the as-received T6 hardness could nearly be reached by artificial aging at 120 °C, regardless of pre-deformation. The maximum deviation from the as-received hardness was 5.8%. The aging time to peak hardness was reduced by nearly 50% when, after the solution heat treatment, the samples were pre-strained at room temperature, in comparison to the undeformed material. For the alloy EN AW-7020, a retrogression heat treatment at 360 °C resulted in a similar cold formability and hardenability. Especially after pre-straining in the quenched state, peak aging could restore the as-received T6 hardness. For the alloy EN AW-7020, retrogression at 360 °C and re-aging with integrated forming is therefore a promising alternative to the solution heat treatment process. Due to the reduced temperature, energy consumption as well as time for heating up the material can be reduced. For the alloy EN AW-7075, solution heat treatment at 480 °C is well suited to restore the as-received T6 hardness, whereas no retrogression and re-aging procedure with sufficient formability and hardenability could be identified.

In order to give definite recommendations for the practical implementation of the W-temper process, further research is required. Obviously, more elaborate aging procedures involving both natural aging and artificial aging at more than one temperature may be applied. In addition, paint baking may be part of the process. Such processes may reduce the overall aging time and could even

### Table 2. Average sizes of the alloy EN AW-7020 in different conditions measured with the linear intercept method.

| Deformation | As-Received | As-Quenched | Peak Aged (27 h) |
|-------------|-------------|-------------|-----------------|
| 0%          | 45          | 59          | 56              |
| 15%         | 56          | 56          | 56              |

Similar observations were made for the alloy EN AW-7075. Therefore, we can exclude any significant effect of the grain structure on the observed changes of the mechanical properties.

### 4. Conclusions

In this work, the potential of W-temper forming is demonstrated for high strength 7xxx series aluminum alloys. The main focus was on the alloy EN AW-7020 and the findings for this alloy were further considered to investigate the alloy EN AW-7075. A general understanding of the effects of various heat treatments in combination with room temperature forming on the artificial aging behavior was developed. The effectivity of the analyzed process variants was evaluated in terms of the softening and ductility increase after the heat treatments, as well as the hardening potential during artificial aging. The hardening potential was determined from a simplified one-step artificial aging treatment. For both alloys, after solution heat treatment, the as-received T6 hardness could nearly be reached by artificial aging at 120 °C, regardless of pre-deformation. The maximum deviation from the as-received hardness was 5.8%. The aging time to peak hardness was reduced by nearly 50% when, after the solution heat treatment, the samples were pre-strained at room temperature, in comparison to the undeformed material. For the alloy EN AW-7020, a retrogression heat treatment at 360 °C resulted in a similar cold formability and hardenability. Especially after pre-straining in the quenched state, peak aging could restore the as-received T6 hardness. For the alloy EN AW-7020, retrogression at 360 °C and re-aging with integrated forming is therefore a promising alternative to the solution heat treatment process. Due to the reduced temperature, energy consumption as well as time for heating up the material can be reduced. For the alloy EN AW-7075, solution heat treatment at 480 °C is well suited to restore the as-received T6 hardness, whereas no retrogression and re-aging procedure with sufficient formability and hardenability could be identified.

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### Figure 10. Optical micrographs of the alloy EN AW-7020. As-received state: (a) view along normal direction (ND), (b) view along transverse direction (TD). Solution heat treated, pre-deformed by 15% and peak aged for 27 h: (c) along ND, (d) along TD.

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result in a higher strength. On the other hand, the long-term behavior of formed and aged parts has to be carefully analyzed to understand the influence of forming on the thermal stability.

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**References**

1. Kaufman, J.G. *Introduction to Aluminum Alloys and Tempers*; ASM International: Metals Park, OH, USA, 2000.
2. Williams, J.C.; Starke, E.A., Jr. Progress in structural materials for aerospace systems. *Acta Mater.* **2003**, *51*, 5775–5799. [CrossRef]
3. Godard, D.; Archambault, P.; Aebly-Gautier, E.; Lapasset, G. Precipitation sequences during quenching of the AA 7010 alloy. *Acta Mater.* **2002**, *50*, 2319–2329. [CrossRef]
4. Azarniya, A.; Taheri, A.K.; Taheri, K.K. Recent advances in ageing of 7xxx series aluminum alloys: A physical metallurgy perspective. *J. Alloys Compd.* **2019**, *781*, 945–983. [CrossRef]
5. Kumar, M. AW-7075-T6 sheet for shock heat treatment forming process. *Trans. Nonferr. Met. Soc. China* **2017**, *27*, 2156–2162. [CrossRef]
6. Viana, F.; Pinto, A.M.P.; Santos, H.M.C.; Lopes, A.B. Retrogression and re-ageing of 7075 aluminium alloy: Microstructural characterization. *J. Mater. Process. Technol.* **1999**, *92*, 54–59. [CrossRef]
7. Zhang, Y.; Milkereit, B.; Kessler, O.; Schick, C.; Rometsch, P.A. Development of continuous cooling precipitation diagrams for aluminium alloys AA7150 and AA7020. *J. Alloys Compd.* **2014**, *584*, 581–589. [CrossRef]
8. Milkereit, B.; Österreich, M.; Schuster, P.; Kirov, G.; Mukeli, E.; Kessler, O. Dissolution and precipitation behavior for hot forming of 7021 and 7075 aluminium alloys. *Metals* **2018**, *8*, 531. [CrossRef]
9. Banhart, J.; Lay, M.D.H.; Chang, C.S.T.; Hill, A.J. Kinetics of natural aging in Al-Mg-Si alloys studied by positron annihilation lifetime spectroscopy. *Phys. Rev. B* **2011**, *83*, 14101. [CrossRef]
10. Deschamps, A.; Livet, F.; Bréchet, Y. Influence of predeformation on ageing in an Al–Zn–Mg alloy—I. Microstructure evolution and mechanical properties. *Acta Mater.* **1998**, *47*, 281–292. [CrossRef]
11. Berg, L.K.; Gjønnes, J.; Hansen, V.; Li, X.Z.; Knutson-Wedel, M.; Schryvers, D.; Wallenberg, L.R. GP-zones in Al–Zn–Mg alloys and their role in artificial aging. *Acta Mater.* **2001**, *49*, 3434–3451. [CrossRef]
12. Li, X.Z.; Hansen, V.; Gjønnes, J.; Wallenberg, L.R. HREM study and structure modeling of the η′ phase, the hardening precipitates in commercial Al–Zn–Mg alloys. *Acta Mater.* **1999**, *47*, 2651–2659. [CrossRef]
13. Sha, G.; Cerezo, A. Early-stage precipitation in Al–Zn–Mg–Cu alloy. *Acta Mater.* **2004**, *52*, 4503–4516. [CrossRef]
14. Mukhopadhyay, A.K.; Yang, Q.B.; Singh, S.R. The influence of zirconium on the early stages of aging of a ternary Al-Zn-Mg alloy. *Acta Metall. Mater.* **1994**, *42*, 3083–3091. [CrossRef]
15. Ibrahim, M.F.; Garza-Elizondo, G.H.; Samuel, A.M.; Samuel, F.H. Optimizing the Heat Treatment of High-Strength 7075-Type Wrought Alloys: A Metallographic Study. *Int. J. Met.* **2016**, *10*, 264–275. [CrossRef]
16. Robinson, J.S.; Tanner, D.A. Residual stress development and relief in high strength aluminium alloys using standard and retrogression thermal treatments. *Mater. Sci. Technol.* **2003**, *19*, 512–518. [CrossRef]
17. Deschamps, A.; Bréchet, Y.; Livet, F. Influence of copper addition on precipitation kinetics and hardening in Al–Zn–Mg alloy. *Mater. Sci. Technol.* **1999**, *15*, 993–1000. [CrossRef]
18. Kovács, Z.; Chinh, N.Q.; Süvegh, K.; Marek, T.; Horváth, G.; Lendvai, J.; Ping, D.H.; Hono, K. The Effect of Cu on Precipitation in Al-Zn-Mg Alloys. In Proceedings of the 9th International Conference on Aluminium Alloys, Brisbane, Australia, 2–5 August 2004; pp. 1192–1197.
19. Cao, C.; Zhang, D.; Wang, X.; Ma, Q.; Zhuang, L.; Zhang, J. Effects of Cu addition on the precipitation hardening response and intergranular corrosion of Al-5.2 Mg-2.0 Zn (wt.%) alloy. *Mater. Charact.* **2016**, *122*, 177–182. [CrossRef]
20. de Argandoña, E.S.; Galdos, L.; Ortubay, R.; Mendiguren, J.; Agirretxe, X. Room temperature forming of AA7075 aluminum alloys: W-temper process. In *Key Engineering Materials*; Trans Tech Publications Ltd.: Stafa-Zurich, Switzerland, 2015; Volume 651, pp. 199–204.
21. ASM Handbook Committee. Heat Treating of Aluminum Alloys. *ASM Handb.* **1991**, *4*, 841–879.
22. Park, J.K.; Ardell, A.J. Effect of retrogression and reaging treatments on the microstructure of Al-7075-T651. *Metall. Mater. Trans. A* **1984**, *15*, 1531–1543. [CrossRef]
23. Richard, D.; Adler, P.N. Calorimetric studies of 7000 series aluminum alloys: I. Matrix precipitate characterization of 7075. *Metall. Trans. A* **1977**, *8*, 1177–1183.
24. Osten, J.; Milkereit, B.; Schick, C.; Kessler, O. Dissolution and precipitation behaviour during continuous heating of Al–Mg–Si alloys in a wide range of heating rates. *Materials* **2015**, *8*, 2830–2848. [CrossRef]
25. Portevin, A.; Le Chatelier, F. Comptes Rendus Acad. Sci. Paris **1923**, *176*, 507–510.
26. Unger, N. Untersuchung einer Thermisch Unterstützten Fertigungskette zur Herstellung Umgeformter Bauteile aus der Höherfesten Aluminiumlegierung EN AW-7020. Ph.D. Thesis, Friedrich-Alexander-Universität, Erlangen-Nürnberg, Germany, 2017.
27. Jung, S.-H.; Lee, J.; Kawasaki, M. Effects of Pre-Strain on the aging behavior of Al 7075 alloy for hot-stamping capability. *Metals* **2018**, *8*, 137. [CrossRef]
28. Jiang, X.J.; Noble, B.; Holme, B.; Waterloo, G.; Tafto, J. Differential scanning calorimetry and electron diffraction investigation on low-temperature aging in Al-Zn-Mg alloys. *Metall. Mater. Trans. A* **2000**, *31*, 339–348. [CrossRef]
29. Czerwinski, F. Thermal Stability of Aluminum Alloys. *Materials* **2020**, *13*, 3441.

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