Periodica Polytechnica Mechanical Engineering, 67(3), pp. 252–258, 2023

Investigation of the Effect of Heat Treatment Time in Case of Recrystallization of Al99.5

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Received: 17 June 2023, Accepted: 23 June 2023, Published online: 30 June 2023

Abstract

In this study the effect of heat treatment time was investigated in case of recrystallization of Al99.5 material samples. Two different type were manufactured based on the previously applied cold forming. 12% and 24% cold forming was applied to the samples before the heat treatment procedure, which was always at 570 °C and cooled in water. Six different heat treatment time were investigated, namely 5, 10, 30, 60, 120 and 240 minutes. After the recrystallization procedure the microstructure and the mechanical properties were determined. It was found that in the case of the 12% cold formed samples after 30-minute-long heat treatment there were still signs of the original microstructure, however it does not affect the mechanical properties. The yield strength and the ultimate tensile strength were independent of the heat treatment time; they were only dependent on the grain size which was expected. A strong dependency can be discovered between the elongation at break and the heat treatment time. A tangent hyperbolic function was fitted on the measured data, which showed that the asymptote was ~29% for both type of samples. This is a 25% increase compared to the 5-minute-long heat treatment time samples, and this value was reached after ~120 minutes. Another result was that the elongation at break dependency on the grain size is decreasing with increasing heat treatment time.

Keywords

aluminum, recrystallization, yield strength, heat treatment time

1 Introduction

Aluminum occupies an extremely important place in industry, as it is used in many places, so we can meet it in, for example, the automotive industry [1], the aviation industry [2], the ship industry [3] and the construction industry [4]. In the case of polycrystalline metals, including aluminum, the size of the grains affects the mechanical properties [5] according to the Hall-Petch equation [6, 7]. This is particularly important, since nowadays there is increasing pressure in the industry to determine the mechanical properties of the given material or equipment from smaller and smaller test specimens.

For these small test specimens, a new test type was developed, namely the small punch test which was later standardized [8]. During this measurement a disk with a diameter of 8 mm and a thickness of 0.5 mm is examined (Fig. 1) [9]. The force – displacement curve is registered, and the mechanical properties of the samples are calculated from it [10, 11]. The proof stress and the ultimate tensile strength can be estimated, but there are a few uncertainties with these calculations. Creep tests can also be done with this measurement setup at high temperatures [9].

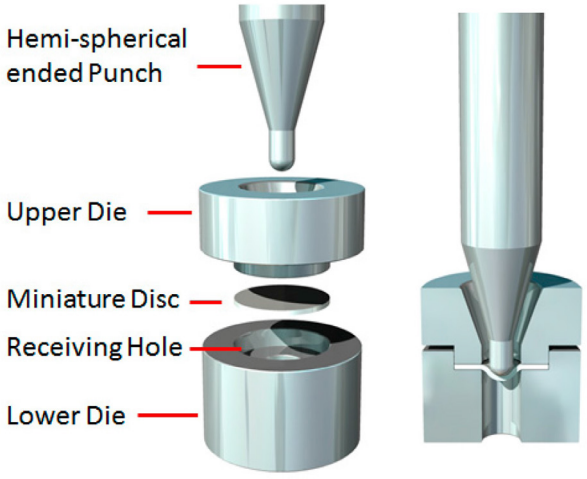

Fig. 1 Schematic representation of the small punch test method adapted from [8]

Cite this article as: Varga, D., Szlancsik, A. "Investigation of the Effect of Heat Treatment Time in Case of Recrystallization of Al99.5", Periodica Polytechnica Mechanical Engineering, 67(3), pp. 252–258, 2023. https://doi.org/10.3311/PPme.22823

The reduction of the size of the test specimens has now reached the point where the traditional Hall-Petch relation is not valid [12, 13]. This is particularly important in the case of recrystallized aluminum specimens, as it is well known that coarse-grained recrystallization can occur (Fig. 2) [14].

During recrystallization, new, stress-free and uniform-sized (i.e., nearly the same size in all directions) grains are formed, which have low dislocation density and are characteristic of the pre-worked state. In other words, during recrystallization, the plastically deformed grains with a large excess of energy do not regain their original state, but completely new, stress-free crystallites are formed. The driving force of recrystallization is actually the energy difference between the deformed and the newly formed grains [5, 14].

Commercially available aluminum, however, contains many impurities that greatly affect recrystallization [15, 16]. The main contaminants of commercially available 99.5% pure aluminum are iron and silicon. Iron is present as particles ranging from one micron to several microns, so it can act as a nucleation site during recrystallization. For example, Fe-FeO [17], Fe-FesC [18], Fe-Si [19] and Al-Si-Cu [20] show such a nucleating property. In addition to the effect on the recrystallization temperature (or time), the increase in the number of nucleation sites can reduce the recrystallized grain size, which was observed in case of Fe-FeO [17], Fe-Si [19], Al-Fe [21] and Al-Si-Cu [20].

The investigation of the recrystallization behavior of cold-rolled aluminum has been a research topic for a long time [22–24]. The mechanism of recrystallization can be inferred based on the recrystallization texture [25]. The initial grain structure significantly affects the recrystallization kinetics, as the preferential nucleation starts at the initial grain boundaries and the increase in the amount of stored energy accelerate the nucleation and growth rate, thereby reducing the recrystallization temperature or time [15].

Hansen and Jensen [26] studied the deformation and recrystallization texture of commercial pure aluminum and found that the rate of texture development for coarse-grained specimens was much lower at small to moderate deformations than for fine-grained specimens.

Zhang et al. [27] investigated AA 5182 aluminum alloy and investigated the effect of initial grain size on recrystallization texture. In their research, they found that the fine-grained alloy recrystallized faster than the coarse-grained alloy because the initial grain boundaries were favored nucleation sites. In addition, they found that the inhomogeneous distribution of nuclei in the coarse-grained alloy led not only to a slow decrease in yield strength and ultimate tensile strength with increasing annealing temperature, but also to significant inhomogeneity of the recrystallized grains.

Vandermeer and Jensen [16] studied the isothermal recrystallization of 90% cold-rolled commercial purity AA1050 aluminum alloy and found that the recrystallization was growth (boundary migration rate) controlled.

During recrystallization, heat treatment parameters also play a decisive role [28–32]. Zhao et al. [33] studied cold-rolled 98% pure aluminum and found that for the ultrafast annealing – only 1 s annealing time – the temperature should be in the range of 410 °C to 435 °C to optimize the mechanical performance of commercial pure aluminum (Fig. 3).

Since our long-term goal is to be able to produce coarse-grained specimens with only a few grains in the examined cross-section, our goal in this article is to find

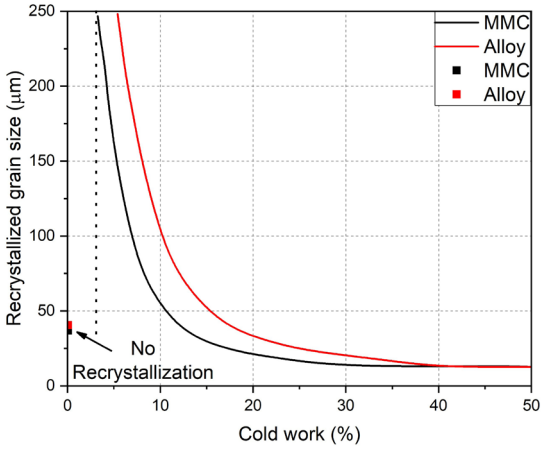

Fig. 2 Recrystallized grain size of the alloy and composite as a function of strain adapted from [14] (Redrawn by Varga and Szlancsik)

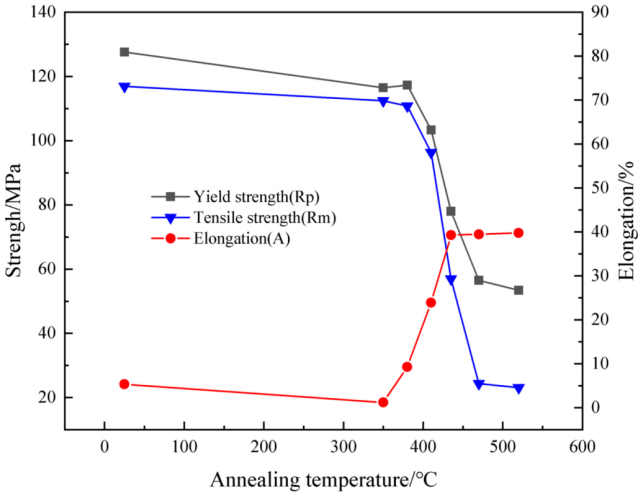

Fig. 3 Mechanical properties of cold-rolled aluminum after ultra-fast annealing at different annealing temperatures [33]

the optimal heat treatment parameters for commercially available aluminum (Al99.5) with which recrystallization can be performed.

2 Materials and methods

During our experiments, an Al99.5 strip was used, the width of which was 10 mm, while the thickness was 1 mm. As a first step, annealing was done at 570 °C for 1 hour, as these strips were made by cold forming, so their formability has already been significantly reduced. After annealing, the samples were deformed by 12% and 24% with an Instron 5965 electromechanical universal material testing machine. Uniaxial tension was used, and then subjected the specimens to recrystallizing heat treatment. In all cases, the heat treatment was carried out at 570 °C and cooled in water, however, the time of the heat treatment was changed, in order to determine the optimal time at which the recrystallization has taken place completely and the residual stresses and dislocation density are also reduced. To this end, six different time length was examined, with 3 samples for every type, the notations for which are listed in Table 1. Also a reference sample type was manufactured without the recrystallization heat treatment.

After the heat treatment, tensile test specimens were machined from the strips, the cross-section of which was 5 × 1 mm. The samples have been etched with aluminum macro-etching solution, the composition of which is included in Table 2.

After etching, the resulting grain structure was examined with an Olympus SZX16 stereomicroscope. After that, tensile tests were performed on the samples at a crosshead speed of 3 mm/min with the Instron 5965 electromechanical universal material testing machine. During the measurement, the force - displacement diagram was registered, from which the engineering stress - engineering strain diagram was calculated. Using these data, the yield strength ($R_{p0.2}$), the ultimate tensile strength (R_m) and the elongation at break (A) were determined.

Table 1 The name of the samples with the heat treatment parameters

Name	Cold forming (%)	Heat treatment time (min)	Name	Cold forming (%)	Heat treatment time (min)
O12	12	0	O24	24	0
R12-5	12	5	R24-5	24	5
R12-10	12	10	R24-10	24	10
R12-20	12	30	R24-20	24	30
R12-60	12	60	R24-60	24	60
R12-120	12	120	R24-120	24	120
R12-240	12	240	R24-240	24	240

Table 2 Composition of the aluminum macro-etching solution

Component	Ratio (%)
Distilled water	18
Nitric acid	16
Hydrofluoric acid	16
Hydrochloric acid (37%)	50

3 Results and discussion

3.1 Microstructure

After the heat treatment and etching, the resulting microstructure was examined (Figs. 4 and 5).

As shown in Figs. 4 and 5, in the case of the 12% cold formed samples 30-minute-long heat treatment was not sufficient for the complete recrystallization to take place, because traces of the original microstructure still can be seen, however in the case of the 24% cold formed samples only 5 minutes were enough to complete the recrystallization. Based on these pictures, it can be concluded that the heat treatment time has to be chosen depending on the previously done cold forming processes to achieve full recrystallization. The resulting grain sizes are 610 ± 165 μm and 238 ± 28 μm in case of the 12% and 24% cold formed samples respectively for every heat treatment time (Fig. 6). The grain sizes were determined based on the stereomicroscopic pictures; the average value was calculated for at least 10 grains in a sample. The original grains are located inside a grain – not in the boundary – which is marked with red circles in Fig. 5.

In Figs. 5 and 6 it can be seen that there are no signs of grain coarsening. It is important, because one can assume that after 240 minutes, grain coarsening will be significant, but the size of the grains did not change during the investigated heat treatment times of the tests.

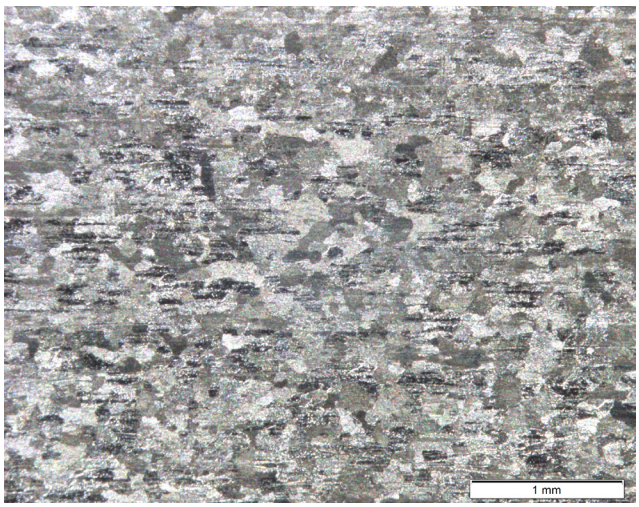

Fig. 4 Original microstructure of the samples (before cold forming and recrystallization)

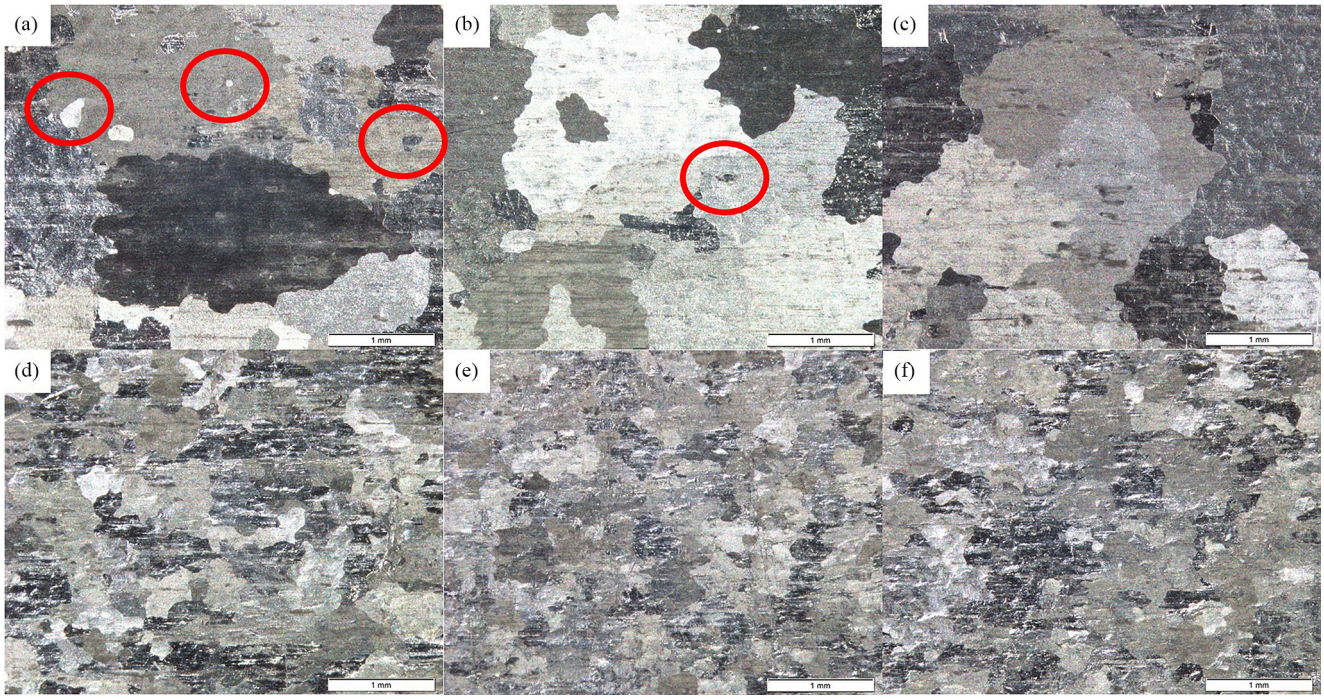

Fig. 5 Microstructure of the 12% cold formed samples after (a) 5 min, (b) 30 min and (c) 240 min with the traces of the original microstructure (red circles), and also the microstructure of the 24% cold formed samples after (d) 5 min, (e) 30 min and (f) 240 min

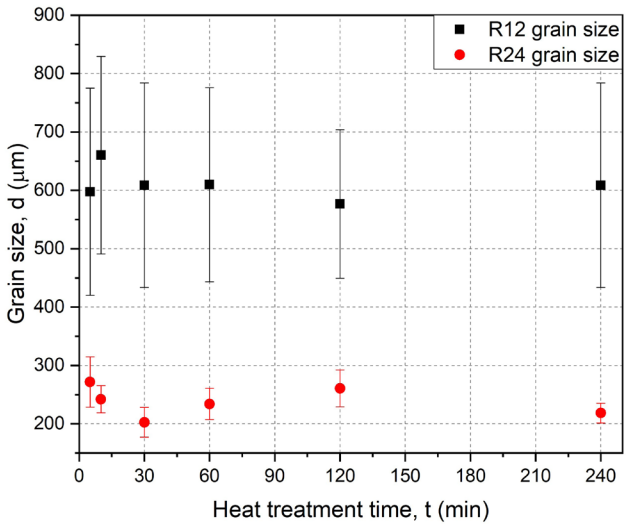

Fig. 6 Grain size after recrystallization as a function of heat treatment time in case of 12% (R12) and 24% (R24) cold formed samples

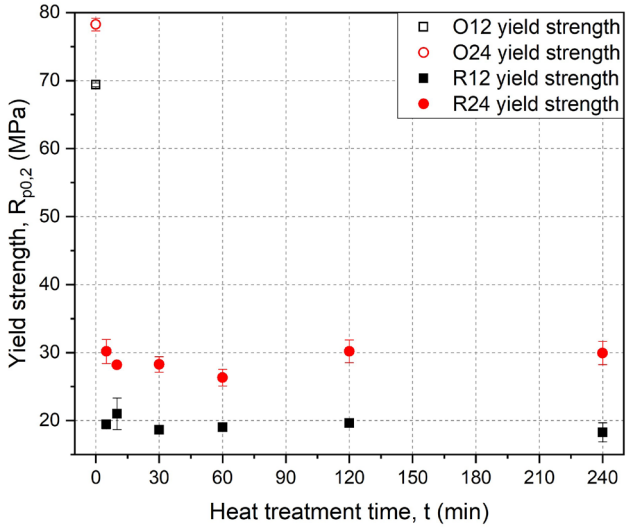

Fig. 7 Yield strength as a function of heat treatment time in case of 12% (R12) and 24% (R24) cold formed samples

3.2 Mechanical properties

The results obtained from the tensile tests are summarized in the diagrams shown in Figs. 7–9.

As expected, the yield strength of the material depends on the grain size of the samples, however there are no signs of grain coarsening which is why the values remain almost the same. It can be stated that the heat treatment time – from 5 minutes to 240 minutes – does not affect the yield strength. In case of the 12% cold formed samples there are no signs of the traces of the original grain structure – which was observed in Fig. 5 – because the yield strength does not decrease significantly. This suggests that the residual stresses completely gone and the dislocation density is very low after only 5 minutes, but grain structure can vary during the heat treatment process.

The ultimate tensile strength depends on the cold forming; however, this dependence is not as significant as in case of the yield strength. The difference was determined for every heat treatment time separately then the average value was calculated. In this case the difference between

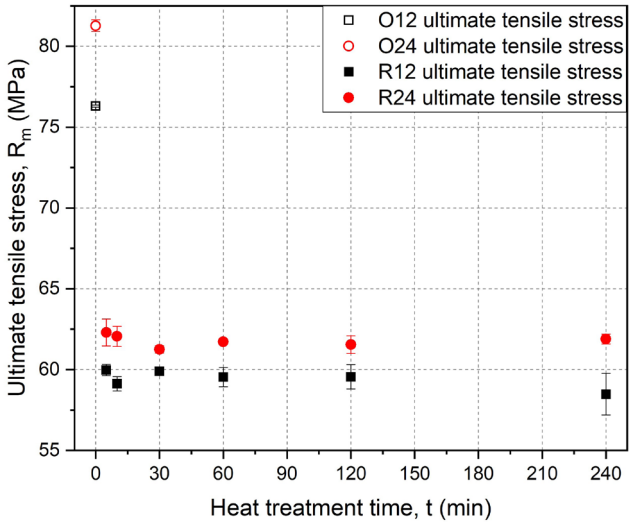

Fig. 8 Ultimate tensile strength as a function of heat treatment time in case of 12% (R12) and 24% (R24) cold formed samples

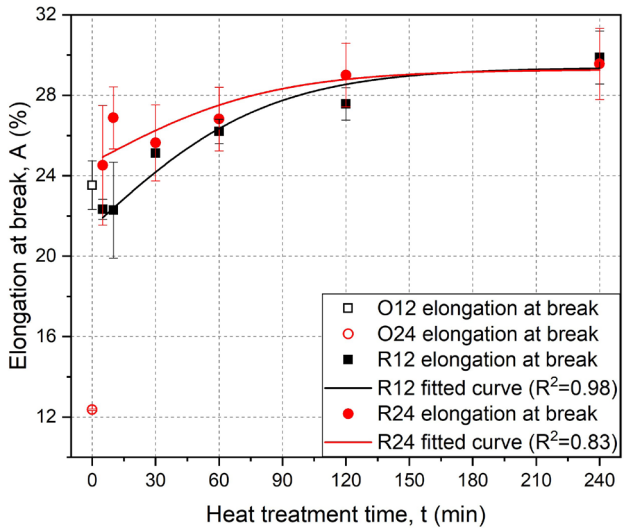

Fig. 9 Elongation at break as a function of heat treatment time in case of 12% (R12) and 24% (R24) cold formed samples

the two sample types is only 4.1% while in case of the yield strength the difference is 37.4%. It can be also seen that the heat treatment time does not affect the ultimate tensile strength, which also supports the statement that there is no grain coarsening during these heat treatments.

The elongation at break depends on the cold forming; however, this dependence disappears for longer heat treatment times. Also as it was expected, without heat treatment this value is really low, and the difference between the two sample types is 12%. The elongation at break has a significant dependence on the heat treatment time. As it can be seen in Fig. 8 the two data sets are overlapping and a curve can be fitted on them in the following form (Eq. (1)):

$$A = a + b \times \tanh(c \times t) \tag{1}$$

where A – elongation at break, a, b and c – parameters and t – heat treatment time. The asymptote value for both cases is ~29%, which is a 25% increase compared to the 5-minute-long heat-treated samples.

This increase is important because the yield strength and ultimate tensile strength did not change significantly during the 240-minute-long heat treatment process, but the elongation at break did, so the material's mechanical properties were improved. This means a tougher material was manufactured with the same strength, which usually not the case, because with higher toughness the strength of the material is decreasing. With these properties this material is more suitable for cold forming applications like deep drawing.

4 Conclusion

Two different previously cold formed (12% and 24%) sample types from Al99.5 were heat treated with different heat treatment times. The following conclusions can be drawn based on the microstructure and the tensile test results.

- There were no signs of grain coarsening after 240 minutes at 570 °C.
- The yield strength and the ultimate tensile strength depend on the grain size; however, they do not depend on heat treatment time within the examined range.
- The elongation at break does not depend on the grain size; however, it is strongly dependent on the heat treatment time. This dependency can be described with a tangent hyperbolic function. The asymptote value was ~29% for both cases which is a 25% increase compared to the 5-minute-long heat-treated samples. This value is reached after ~120 minutes.
- The optimal heat treatment time is 120 minutes at 570 °C.

Acknowledgement

Project no. TKP-6-6/PALY-2021 has been implemented with the support provided by the Ministry of Culture and Innovation of Hungary from the National Research, Development and Innovation Fund, financed under the TKP2021-NVA funding scheme. This research was partly supported by the János Bolyai Research Scholarship of the Hungarian Academy of Sciences No. BO/00442/22/6.

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