Softening Behavior During Annealing of Overaged and Cold-rolled Aluminum Alloy 7075

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This work proposes studying the softening kinetics of aluminum alloy 7075-T6. Firstly, AA 7075 strips were overaged (300°C and 5 hours). Then, the overaged strips were cold-rolled at room temperature (45% in thickness reduction) and, after that, annealed at different temperatures (200°C and 250°C) and time intervals (30 minutes - 4 hours). By using polarized light microscopy, EBSD and Vickers hardness measurements, the softening mechanisms were determined and proper mathematical models (Kuhlmann, Johnson-Mehl-Avrami-Kolmogorov and Austin-Rickett) were used to analyse the experimental data. The results show that although almost all observed softening can be attributed to recovery, the phenomena is well described by using mathematical models for phase transformations (JMAK and Austin Riccket). In order to promote recrystallization of overaged AA 7075, an additional thickness reduction (75%) were used. These samples were annealed isocronically during 1 hour (100 - 400°C) and it was found that recrystallization only took place at 400°C.

Keywords: *Aluminum alloy 7075, annealing, cold-rolling, recovery, recrystallization.*

1. Introduction

The 7000 series of commercial aluminum alloys are widely used in structures that require a high strength/weight ratio, like aircrafts structures. These alloys undergo thermo-mechanical treatments that provide a microstructure with fine, coherent and/or semi-coherent, and well distributed $MgZn_2$ precipitates, which are the mainly responsible for alloy's high strength (Yield stress ≈ 500 MPa). Although the 7075 aluminum alloy has high mechanical strength, its low toughness and stress-corrosion susceptibility might be a problem for some applications. Therefore, new thermo-mechanical cycles have been developed to overcome these difficulties [1,2].

The grain refining provided by recrystallization is desirable for most metallic materials, since it leads to an increase in both mechanical strength and toughness [3]. Therefore, the knowledge about AA 7075 recrystallization kinetics under different conditions is useful to reach desired microstructures and properties.

In the present study, the softening behavior of AA 7075 - T6 (Al - Zn - Mg - Cu) was investigated after overaging (300°C - 5 hours), cold-rolling (45% and 75%), and subsequent annealing.

2. Material and Experimental Procedure

All the samples used in this work were obtained from a 10 mm thick rolled plate of AA 7075 - T6 alloy. The alloy's chemical composition is presented in Table 1.

The experimental procedure can be summarized in three main steps: a) As received AA 7075 (T6) was initially characterized by using polarized light microscopy (PLM), scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), X-ray diffraction (XRD), transmission electron microscopy (TEM) and Vickers hardness test (HV 0.3); b) The AA 7075 (T6) was overaged at 300°C during 5 hours in a muffle furnace and quenched in water. After that, the overaged samples underwent SEM, XRD, TEM and Vickers hardness test again. Then, overaged strips were cold-rolled from a thickness of 10 mm to 5.5 mm and 2.5 mm (thickness reductions of 45% and 75%); c) The overaged and cold-rolled samples underwent isochronal and isothermal annealing heat treatments. Vickers hardness measurements from all annealed samples were obtained and the softening kinetics was studied by using these hardness data.
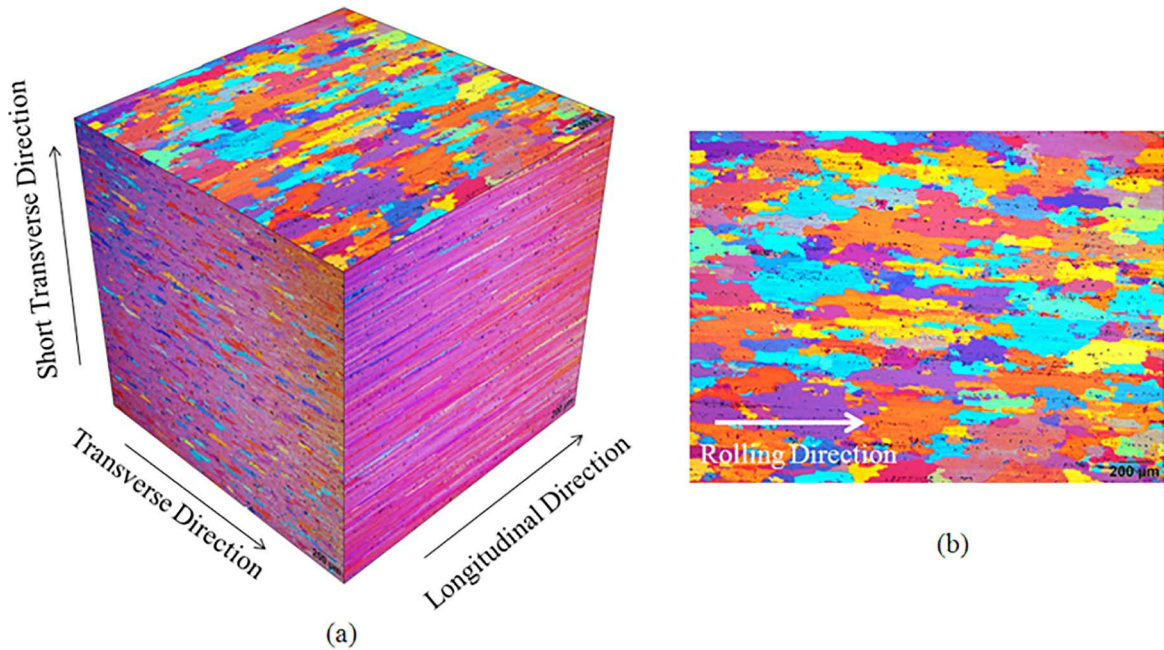
3. Results and Discussion

AA 7075 -T6 microstructure obtained by using PLM is presented in Figure 1. It is possible to notice elongated grains in the rolling direction.

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Table 1. Chemical composition (mass %) of AA 7075 used in the present work.

Component	Ti	Mn	Si	Cr	Fe	Cu	Mg	Zn	Al
% mass	0.02	0.08	0.08	0.19	0.26	1.3	2.3	5.2	90.57

Figure 1. Microstructures of AA 7075 obtained by using polarized light microscopy. (a) views of three different planes and (b) a view of the rolling plane.

The grain boundaries show an undulated geometry. It may happen due to the high concentration of second phase precipitates that difficult the boundaries mobility during recrystallization process. The work of Martorano et al. calls those grain structures protrusions, and concludes that these undulations are caused due to a non -uniform strain energy distribution [4]. Similar grain structures are observed in other AA 7075 studies [5,6].

Analyses made by SEM identified the presence of precipitates (~10µm), aligned with the rolling direction. Figure 2 shows one of those precipitates and its EDS analysis results.

The observed particles contain, according to the EDS analysis, high contents of aluminum and copper, as well as magnesium and iron. It indicates that these precipitates are possibly a phase like Al_2MgCu or Al_7Cu_2Fe. The Al_2CuMg phase was found by Cogan [7] while an AlCuMg phase was reported in Wert's [8] studies. These intermetallic particles are insoluble in Al matrix and do not have strong influence on softening during annealing (by recrystallization).

Conventional precipitation hardening (T6) leads to a fine and metastable dispersion of $MgZn_2$ particles that cannot be seen at the magnification of the micrograph shown in

Figure 3. The observed particles are insoluble dispersoids formed by Cr, as indicated by the high Cr content found via EDS analysis.

After the overaging heat treatment (300°C / 5 hours), small particles can be seen along grain boundaries, as shown in Figure 4.

Apparently, those particles are $MgZn_2$ coarsened precipitates, since it is not possible to observe them in AA 7075 - T6 samples. An EDS analysis indicates that the particles shown in TEM micrograph of Figure 5 are $MgZn_2$.

Figure 6 presents the microstructure obtained from polarized light microscopy of an overaged and cold-rolled (45% in thickness reduction) sample.

The grains of rolled samples are elongated along the rolling direction and their colors are not well defined as those in AA 7075 - T6 condition (compare it to Figure 1). The rolling step certainly changes the density and distribution of dislocations and leads to orientation changes inside grains, which result in the mixed color effect under polarized light [9].

Table 2 presents Vickers hardness measurements of T6, overaged and cold-rolled (after overaging) samples.

T6 condition shows the highest hardness, since its microstructure is composed of nanometric $MgZn_2$ particles,
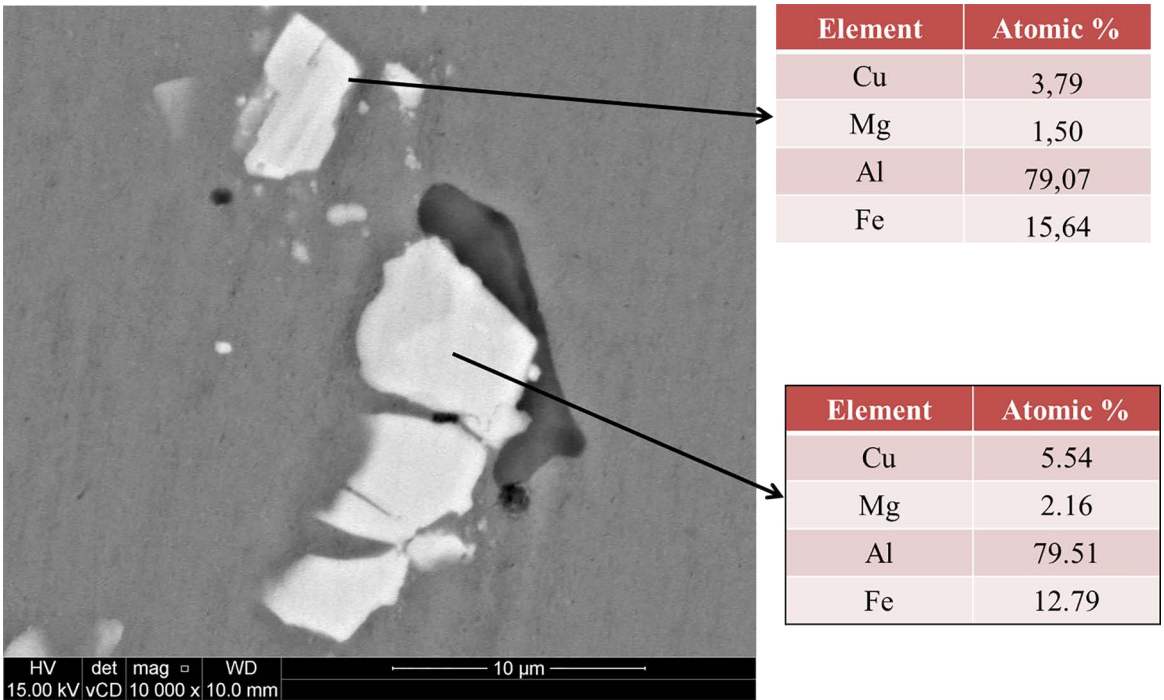
Element	Atomic %
Cu	3,79
Mg	1,50
Al	79,07
Fe	15,64

Element	Atomic %
Cu	5.54
Mg	2.16
Al	79.51
Fe	12.79

Figure 2. SEM image of observed particles.

Element	Atomic %
Cu	3.00
Mg	1,50
Al	85.26
Cr	1,89
Zn	2,89
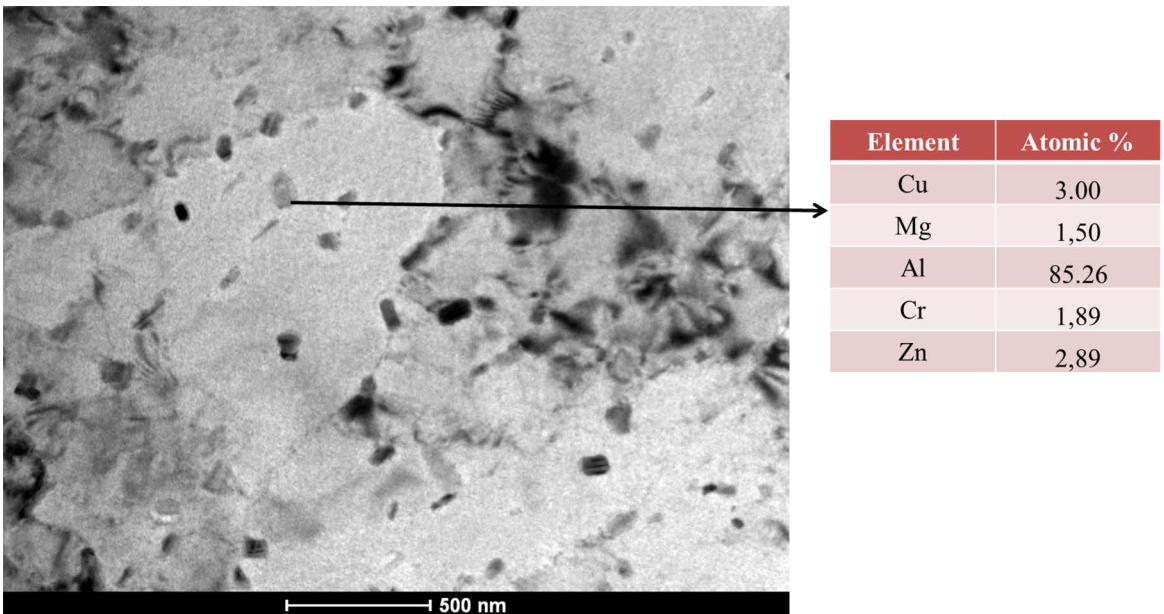
Figure 3. TEM micrograph of AA 7075 (T6). The observed particles are insoluble dispersoids and they are constituted of Cr.

which are responsible for making the dislocation motion difficult. As these particles grow during the overaging step, they lose their coherence with Al matrix and, therefore, lose efficiency in anchoring dislocation lines. Consequently, the hardness decreases.

As the material is cold-rolled, it becomes saturated with new dislocations, which increases hardness and the resistance to plastic deformation due to the interaction among these line defects.

After cold-rolling, the samples were annealed. Firstly, 4 hours isochronal heat treatments were carried out between 150 and 300ºC. Results are presented in Figure 7.

It is possible to notice that the hardness sharply decreases around 200°C, while no significant changes occur for 150°C. On the other hand, at 300ºC the softening was relatively fast. This isochronal curve is relevant for planning the next isothermal heat treatments.

Overaged and cold-rolled samples were submitted to two isothermal heat treatments at 200 and 250°C. Results for both annealings are shown in Figure 8. After 4 hours, the sample treated at 250°C reaches the hardness level of overaged material (81 HV), whilst samples heat treated at 200°C did not reach such softening degree (96 HV).

In order to identify the softening mechanisms, annealed samples were analyzed via EBSD. The results for the sample annealed at 250°C (4 hours) are shown in Figure 9. A large quantity of low-angle grain boundaries (red lines) may be seen inside regions delimited by high-angle grain boundaries (blue lines), probably the original grains (those existing before the samples were cold-rolled). It is also possible
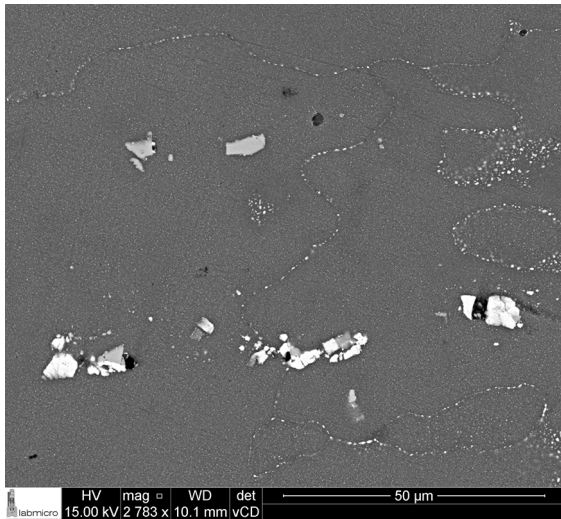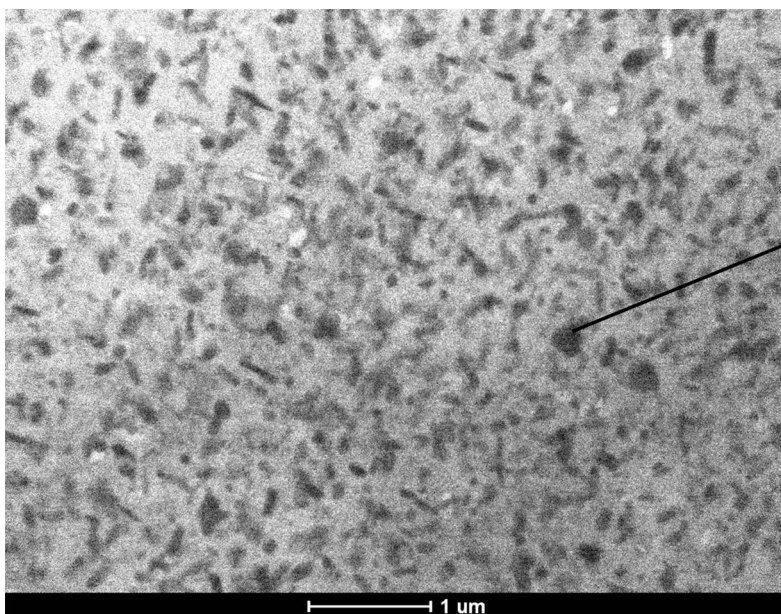
to see few and small grains, probably new recrystallized grains. Since the volume fraction of recrystallized grains is smaller than 5%, the decrease in hardness after annealing can be explained by intense recovery.

A model proposed by Kuhlmann [10] to describe the recovery kinetics was used. This model states that the percentage
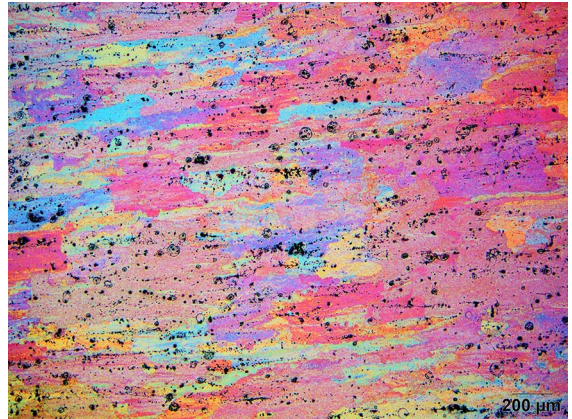

Figure 6. Microstructure of AA 7075 (rolling plane) after cold-rolling obtained by using polarized light microscopy.

Table 2. Vickers hardness measures of AA 7075 at different conditions.

Themomechanical treatment	Vickers Hardness
T6 (starting condition)	180 ± 8
Overaged (300°C – 5hours)	82 ± 2
Overaged and then cold-rolled (45%)	118 ± 4

Figure 4. SEM image showing small particles of $MgZn_2$ along matrix grain boundaries.

Element	Atomic %
Cu	0.80
Mg	3.42
Al	90.92
Fe	0.34
Zn	4,83

Figure 5. TEM micrograph showing $MgZn_2$ coarsening and particles volume fraction increasing after AA 7075 overaging (300°C and 5 hours).

recovery (X) of deformed and annealed metallic materials can be expressed as:
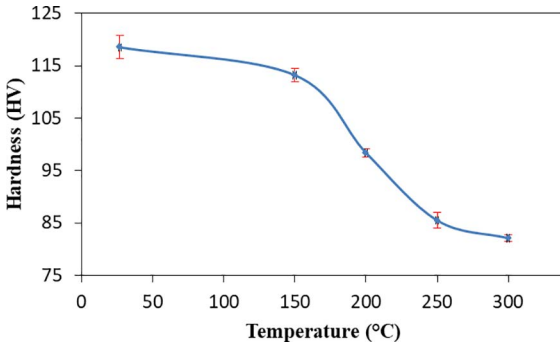
$$X = A - B \ln t \qquad (1)$$

Figure 7. Softening behavior of cold - rolled (45%) samples annealed for 4 hours at different temperatures
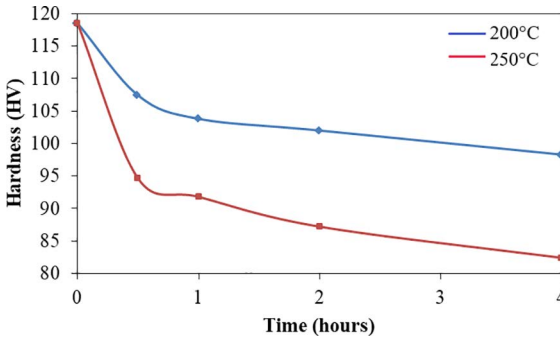

Figure 8. Softening behavior of samples annealed at 200°C and 250°C.

Where "A" and "B" are constants to be determined and "t" is the annealing time. The percentage of recovery can be estimated from hardness data according to:

$$\%Re\,\mathrm{cov}\,ery = \frac{H - H_X}{H - H_R} \qquad (2)$$

where H is alloy original hardness in the cold-rolled condition; H_X is hardness after annealing under a specific condition and H_R is hardness for a sample that has been fully recovered. The use of mechanical properties, such as hardness and tensile yield strength, to study microstructural changes has also been adopted by some authors. Tajally and Huda [11] used a similar model to study the recrystallization kinetics for AA 7075. Sarkar [12] found good correlation between mechanical properties (tensile yield strength) and quantitative metallography by applying a similar method in studies of AA 5754 recrystallization. Figure 10 illustrates the data fitting for both annealing temperatures, 200°C and 250°C.

Alternatively, the results were fit to two other models, Austin-Rickett (AR) [13] and Johnson-Mehl-Avrami-Kolmogorov (JMAK) [14] Both models are traditionally used to describe phase transformations kinetics. The AR formula is expressed by:

$$Log\left(\frac{f}{1-f}\right) = k \log(t) + C \qquad (3)$$

where "f" is transformed fraction, "t" is annealing time and "k" and "C" are constants to be determined experimentally. Figure 11 shows the results of AR data fitting for both 200°C and 250°C.
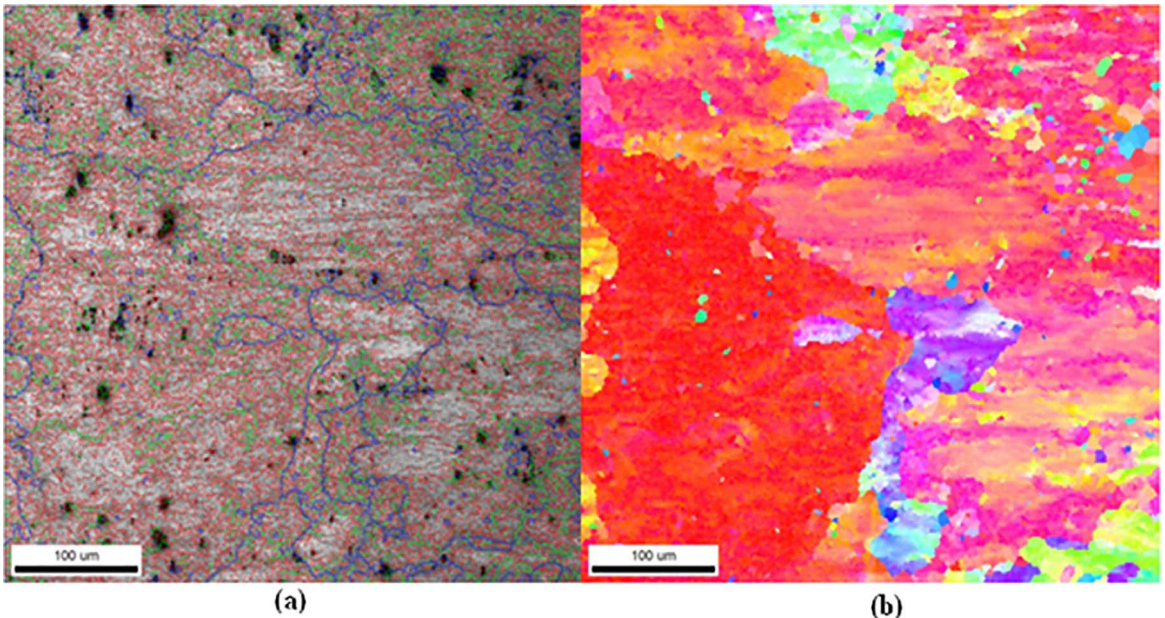

Figure 9. EBSD results for sample treated at 250°C for 4 hours. (a) Grain boundaries map. The red lines represent low-angle grain boundaries ($\theta < 5°$), whilst green and blue lines represent medium ($5° < \theta < 15°$) and high ($\theta > 15°$) angle grain boundaries, respectively. (b) EBSD map showing only few new recrystallized grains.
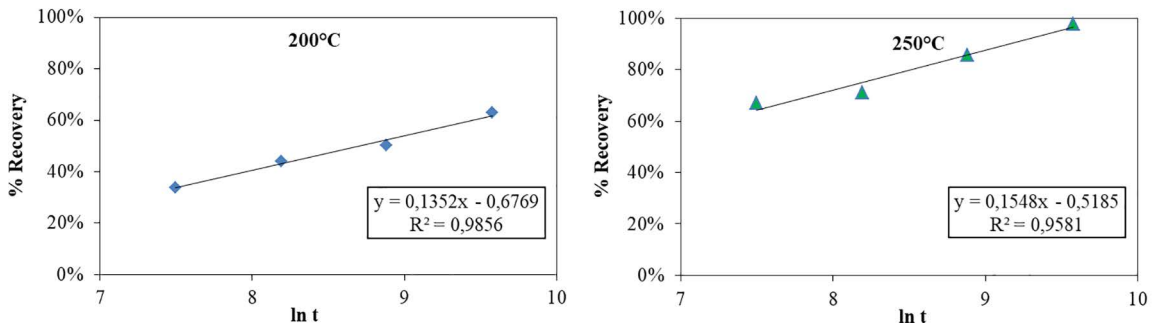
Figure 10. Variation of %Recovery with isothermal annealing time (t) at 200°C and 250°C.
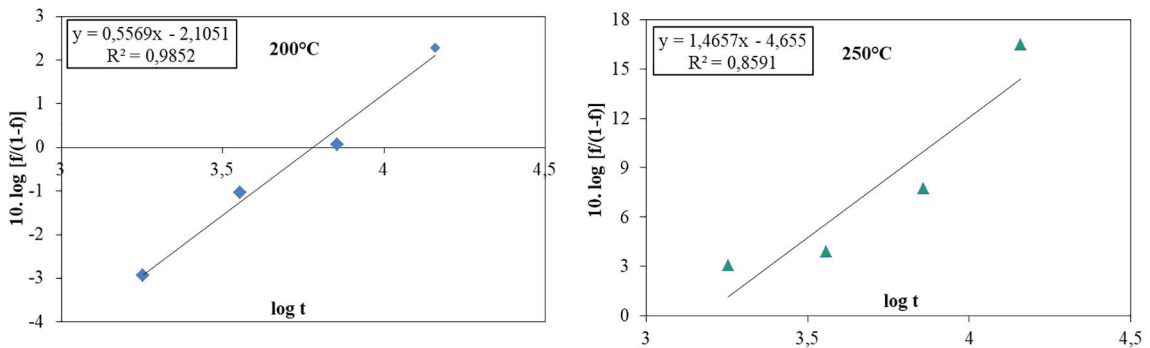

Figure 11. Austin-Riccket's data fitting of isothermal heat treatments at 200°C and 250°C.

The JMAK relation can be expressed as:

$$f = 1 - \exp(-Dt^n) \qquad (4)$$

Where "f" is recrystallized grains' volume fraction, "t" is annealing time and "B" and "n" are constants to be determined experimentally. It is possible to express JMAK equation by using a traditional linear form:

$$\ln\left[\ln\frac{1}{(1-f)}\right] = -\ln D + n \ln t \qquad (5)$$

Figure 12 presents the results JMAK plots (both linear and not linear form) for both annealing temperatures, 200°C and 250°C.

Both equations fit well to experimental hardness data, although no evidences of recrystallization are found in any annealed sample. Regarding JMAK's and Austin-Rickett adjusts; they represent a really interesting, curious and unexpected outcome. Additional studies by using more annealing conditions may clarify why those models, based on nucleation and growth, have described recover kinetics so well. All adjustment data are summarized in Table 3.

It may be noticed that the best result (in terms of R^2) is obtained when the model proposed by Kulhmann is used. Additionaly, it is also possible to realize that all models seem to work better at lower annealing temperatures.

In order to obtain recrystallized samples, a new condition of thickness reduction was used (75% in thickness reduction)

for providing a higher level of strain energy and, consequently, a higher driving force for recrystallization. This time, an initial 1 hour isocronical annealing was performed. The results are shown in Figure 13. Again, there is a sharply decrease in hardness for temperatures between 150°C and 200°C. Nevertheless, micrographs obtained by light polarized microscopy do not show new recrystallized grains for samples annealed within 150°C and 350°C.

For 75% reduction of thickness, recrystallization phenomena is only observed for 400°C after 1 hour, as shown in Figure 14. For these annealing conditions, the microstructure is all composed of new equiaxed grains.

The temperature (400°C) were kept, and three isothermal annealings were performed (8, 15 and 30 minutes) by using overaged and cold-rolled (75% in thickness reduction) samples. The micrographs for these annealed samples are shown in Figure 15. Samples annealed for 15 and 30 minutes are fully recrystallized, whilst the one annealed for 8 minutes seems to be only recovered. Apparently, recrystallization of samples which underwent 75% of thickness reduction takes place for time intervals between 8 and 15 minutes. It is very difficult, thus, to study recrystallization kinetics by using quantitative stereology methods, since the whole process takes place quickly.

The obtained results will be briefly summarized. AA 7075 at the starting condition (T6) has high hardness (180 HV) due to nanometric $MgZn_2$ particles distributed in aluminum solid solution matrix. Since this alloy in as-supplied condition shows
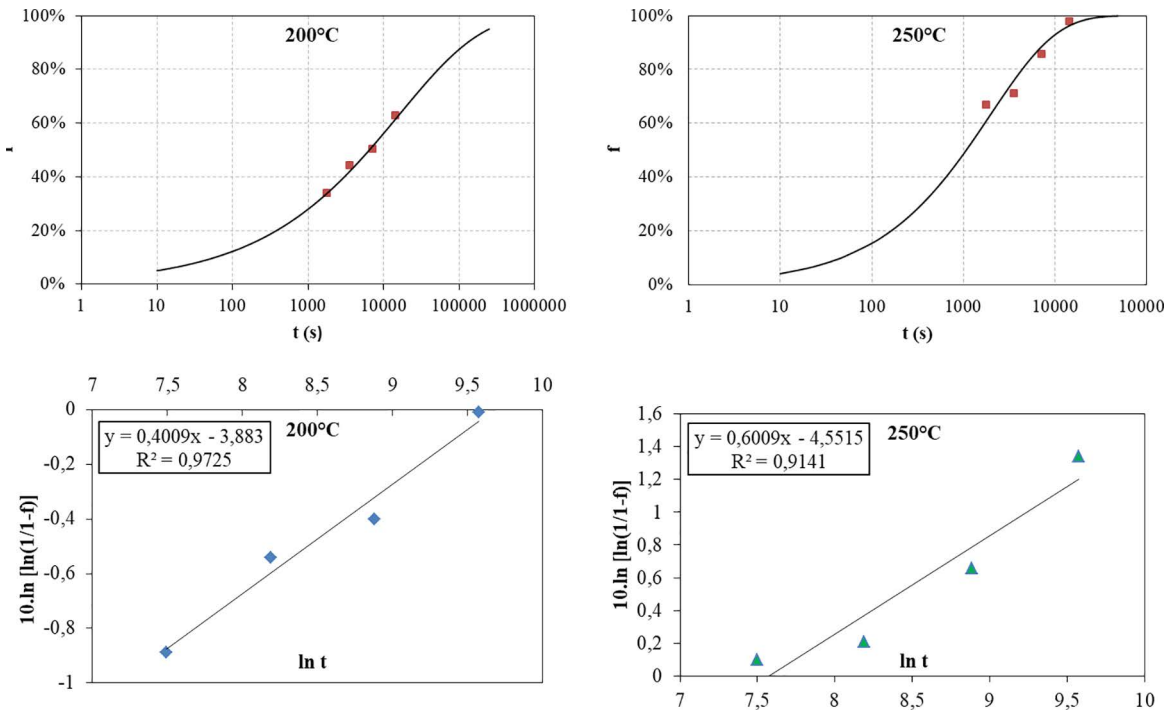
Figure 12. JMAK's exponential and linear data fitting of isothermal annealings at 200°C and 250°C.

Table 3. Summarized results of all models used to adjust the experimental data

Kulhmann	A	B	R^2
200°C	0,677	0,135	0,9856
250°C	-0,52	0,155	0,9581
Austin - Riccket	**k**	**C**	R^2
200°C	0,557	-2,105	0,9852
250°C	1,466	-4,655	0,8591
JMAK	**n**	**D**	R^2
200°C	0,4	0,021	0,9725
250°C	0,6	0,011	0,9141

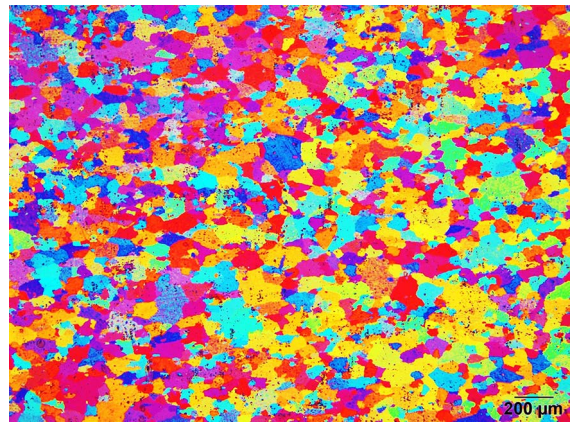**Figure 14.** Microstructure of overaged AA 7075 after cold-rolling (75% in thickness reduction) and annealing (400°C and 1 hour). Obtained by using polarized light microscopy.

Figure 13. Softening behavior of cold-rolled (75%) samples annealed for 1 hour at different temperatures.

low ductility, which makes cold-rolling in T6 condition very difficult, it was overaged at 300°C for 5 hours, resulting in coarsening of $MgZn_2$ particles and, consequently, in hardness decreasing (82 HV). After overaging step, $MgZn_2$ particles can be observed by transmission electron microscopy and also by scanning electron microscopy (Figures 4 and 5).

Cold-rolling induces dislocations arrays and changes the shape of grains, as shown in Figure 6. The Vickers hardness increases from 82 HV to 118 HV due to work hardening.
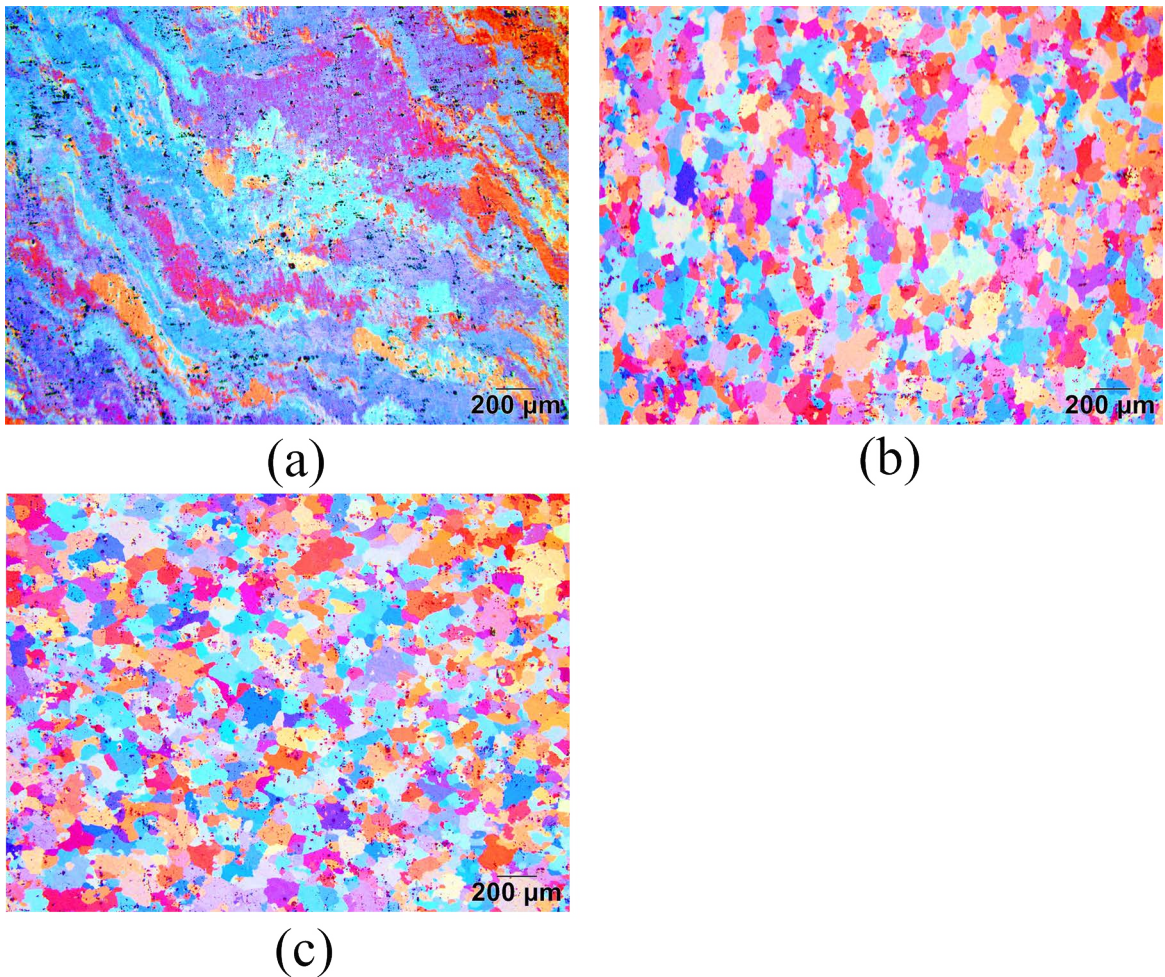
(a)

(b)

(c)

Figure 15. Microstructure of overaged AA 7075 after cold-rolling (75% in thickness reduction) and annealing (400°C) for (a) 8 minutes, (b) 15 minutes and (c) 30 minutes. Obtained by using polarized light microscopy.

Subsequent annealing results in hardness decrease, as shown in Figures 7 and 8. Since no new recrystallized grains are found by optical microscopy and EBSD maps shows only a very low fraction of new recrystallized grains, the main contribution to the observed softening is attributed to recovery mechanisms. This assumption is confirmed when a classic model proposed by Kuhlmann to study recovery kinetics is applied. This model shows good agreement with experimental data (Figure 10). The good correlation to JMAK and Austin-Rickett (AR) models is unexpected, since these models are not proper for describing recovery kinetics.

By using a higher degree of thickness reduction (75%), it is found that for 1 hour annealing, new recrystallized grains are not obtained until the temperature of 350°C. For 400°C annealing, samples become fully recrystallized quickly (a time period between 8 and 15 minutes), difficulting thus the study of recrystallization kinetics.

4. Conclusions

1. Isothermal kinetics of softening can be described both by a classical equation (Kuhlmann equation) used for recovering kinetics and by two equations frequently used for nucleation and growth phase transformations such recrystallization (JMAK equation).

2. The degree of reduction of 45% is not enough to promote a significant recrystallized volume fraction for overaged samples of AA 7075, as it is possible to observe via optical microscopy using polarized light and by EBSD analysis. Therefore it is possible to state that, at least for the annealing temperatures used in this work, the critical strain for recrystallization is higher than 45% in thickness reduction.

3. The model proposed by Kuhlmann [10] (and frequently utilized to describe the recovery kinetics [15]) fits very well to hardness measurements. In this case, experimental data shows good correlation to the mathematical model, and almost all softening can be attributed to recovery mechanisms. Unexpectedly, JMAK and AR models also fit the experimental data well.

4. The use of 75% of reduction is not enough to promote recrystallization for annealing temperatures between 150 and 350 °C (1 hour). By using 400°C it is possible to achieve a microstructure composed of recrystallized grains for different annealing times. A fully recrystallized microstructure for overaged and 75% cold-rolled samples is achieved (at 400°C) in a period of time between 8 and 15 minutes.

Although it was not possible to enhance the material mechanical properties, the present study has achieved interesting results about recovering kinetics and also a grain refinement route. It was found that mathematical models used to quantify recrystallization kinetics can (at least for this specific situation) describe recovery kinetics fairly well.

Future works may aim in achieving mechanical properties enhance by using, for instance, a combination of work hardening and precipitation in 7XXX aluminum alloys (in this case, the main idea is to solubilize, strain and carry out aging treatments at temperatures that are low enough for not causing recrystallization.

5. Acknowledgements

6. References

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