

## RESEARCH PAPER

## PRECIPITATION STATE OF WARM WORKED AA7050 ALLOY: EFFECT ON TOUGHNESS BEHAVIOR

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## ABSTRACT

Usually, strength-toughness combination in aluminum alloys is improved by heat treatment (solid solution followed by quenching and reheating) after a deformation process at high temperature. In some cases, a cold working step is added in the manufacturing process before heat treatment aimed to enhance the alloy strength. In recent time, some trials have been carried out finalized to replace the cold working step with a warm deformation. Such a process route appeared to be quite effective in improving the toughness behavior of 7xxx alloys. Anyway, a metallurgical explanation for such behavior has not still been reported. In this, a comparison of the precipitation state following the two different routes is reported. Results show clear differences in the nanoprecipitation densities in the two cases, claiming for their responsibility in the definition of the toughness behavior.

**Keywords:** toughness; aluminum alloys; heat treatment; precipitation

## INTRODUCTION

Light alloys were considered for a long time the best solution for most of the high-tech applications, including sport equipment [1], energy and automotive [2]. Aeronautical industry was considered one mostly requiring such alloys properties. Among lightweight alloys, the aluminum based ones are assuming industrial impact following their specific combination of properties [3]. Following to that, they seem to be the most promising candidates for structural aerospace designers if compared with different alloys [4-6]. Their specific behavior is known to depend on alloying strategies and processes, as reported in a rich literature (e.g. [7-9]). The choice of alloying elements is made based on the their individual and synergic impact on microstructure and hence on mechanical behavior. The above statement is reported in detail in [10]. Savage and colleagues [11] described the effect of Cu addition on the hardening of Al-Mg-Si alloys characterized by a ultra-fine grain. In the large number of aluminum alloys the AA7050 one assumes particular importance, following its specific balance between tensile properties and corrosion resistance [12, 14]. Such quite promising behavior is achieved by proper recrystallization phenomena occurring during and after the hot deformation stage [14-17]. Physically based constitutive equations and their capability to predict the microstructural evolution in such alloy are reported in [18]. A novel approach to recrystallization phenomena in aluminum alloys is proposed in [19], to be applied in the case of complex geometry parts, commonly produced by closed-due forging and solution and quenching heat treatment. Such effect is also reported in detail by Mac Kenzie in [20].

AMS 4333 International Standard calls for an intermediate cold working process step, with a maximum 5% allowed cold upsetting, to be performed before the two ageing final steps, after the solution heat treatment. This with the aim to best

define the precipitation state (in terms of precipitation size distribution) so assuring the best achievable mechanical properties combination [21]. Concerning this topic, Wyss et al. proposed the US Patent [22], showing beneficial effect due to an intermediate warm hardening process step, to be executed instead of the standard cold upsetting, on the fracture toughness properties. Such process route appeared to be quite promising in the case of 7xxx alloys: in particular an improvement of toughness was found without any deterioration of hardness and tensile behavior [23]. Such route also allowed improving the component homogeneity by means of grain refinement [24]. This was explained in terms of dislocation cross-slip during deformation at the involved temperature range in the considered process. This phenomenon allows a grain re-orientation with a consequent re-organization of sub-grains, moving towards high angle boundaries: the higher the deformation temperature, the easier the process [25]. Such phenomena are reported in literature as Continuous Dynamic Recrystallization (CDR) [26-28]. Other phenomena, such as strain hardening [29, 30] and recovery [31, 32], depend on dislocation evolution in dependence on other present crystallographic imperfections. In this paper the effect of process routes on the precipitation size distribution is reported.

## MATERIAL AND METHODS

The AA 7025 alloy nominal composition is reported in **Table 1**.

**Table 1** Chemical analysis of AISI 441 (main elements, mass %).

Elements	Al	Cu	Mg	Zn
Wt. %	balance	2.3	2.2	6.25

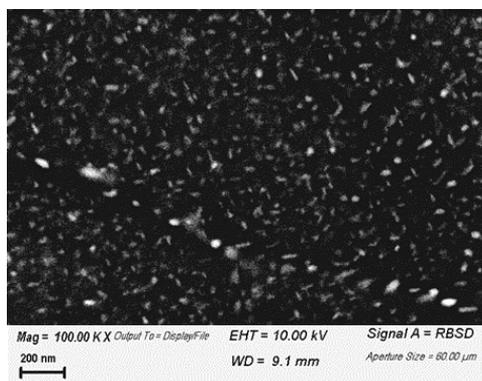
Three families 10 cm x 6 cm x 3 cm specimens A, (specimens A1, A2 and A3), B (specimens B1, B2 and B3) and C (speci-

mens C1, C2 and C3) were manufactured from around a hot forged bar, characterized by an initial diameter  $\Phi = 120$  mm. The heat treatment satisfied the AMS2770N specification requirements. The process was completed with room temperature up-setting and final two stages aging at 5 h at 394 K + 8 h at 450 K (samples A). The two innovative cycles (samples B and C) just differed from AMS2770N specification requirements in terms of up-setting temperature: as a matter of fact, they were carried out at 423 K and 473 K instead of room temperature. All the other cycle steps were unmodified (Table 2).

**Table 2** Heat treatment conditions

Specimen	Solution heat treatment	Water Quenching	Deformation temperature (K)	First ageing step	Second ageing step
A	YES	YES	293	YES	YES
B	YES	YES	423	YES	YES
C	YES	YES	473	YES	YES

All specimens underwent a solution heat treatment at  $T=748$  K for 5 h, water quenching, 5% warm deformation, ageing. The process conditions differ on the upsetting temperature, (see Table 2). A1, A2 and A3 samples were deformed at room temperature; samples B1, B2 and B3 at 423 K and samples; C1, C2 and C3 at 473 K. After heat treatment transverse specimens (in agreement with ASTM-E399) were machined and tests KIC toughness were performed (according to the ASTM E399 standard). Specimens machined starting from the three groups (A, B and C) were prepared for metallographic examination. Grain size was measured by light microscopy (LM) according to ASTM E112 specification. Precipitation state analysis was performed with a scanning electronic microscope SEM-FEG (SEM FEG LEO 1550 ZEISS (McQuairie, London, UK) equipped with an EDS OXFORD X ACT system (v2.2, Abington, UK). Precipitates number counting has been performed by means of IMAGE-J Fiji 1.46, a software for the automatic images processing and analyses program. The image analysis was carried out by setting a Feret-diameter threshold of 10 nm. An example of SEM FEG image prepared for precipitation number count by mean of IMAGE-J Fiji 1.46 software is reported in Figure 1.

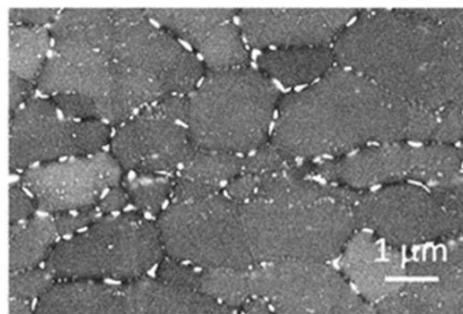


**Fig. 1** Example of the SEM-FEG image (specimen A)

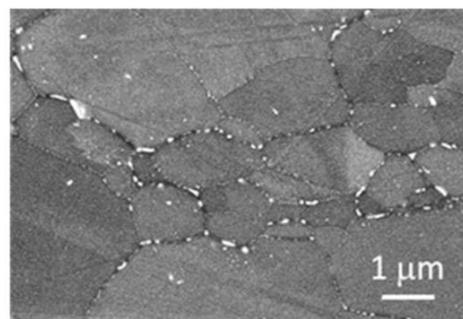
## RESULTS AND DISCUSSION

The effect of the up-setting temperature increase on microstructure is shown in Figures 2 and 3: results clearly show a grain size refinement. Results from mechanical properties

related to the considered materials are reported in detail in [33] and are here summarized for the reader (see Table 3).



**Fig. 2** Sample A microstructure (up-setting T: 293 K)



**Fig. 3** Sample B microstructure (up-setting T: 423 K)

**Table 3** Mechanical properties and grain size evolution with intermediate up-setting temperature

Specimen	Upsetting temperature (K)	HB	YS (MPa)	K <sub>IC</sub> (MPa m <sup>1/2</sup> )	Grain size (μm)
A	293	145	450	26.8	10.1
B	423	147	455	28.3	8.6
C	473	152	470	30.1	7.5

In particular, results reported in put in evidence a not negligible K<sub>IC</sub> improvement, even if both Brinell hardness and yield strength were increased. It is worth to be noted that an up-setting increase up to 473 K resulted in a 10% toughness improvement in terms of K<sub>IC</sub>, with respect to material processed in standard condition. Table 3 also shows that tensile properties were more sensitive to up-setting temperature than hardness: this suggests a significant fine precipitation variation mainly following to Guiner-Preston zones [34, 35] formation acting as a barrier to grain size evolution. The precipitation distribution evolution with up-setting temperature was analyzed by image analysis.

Microstructures of samples A1, B1 and C1 as obtained by SEM-FEG at high magnification are shown in Figure 4, Figure 5 and Figure 6 respectively.

Such analysis allowed to divide precipitates in the following families:

1. larger precipitates located at grain and sub-grain boundaries (size ranging=100-500 nm);
2. fine precipitates inside grains and sub-grains (size ranging=20-100 nm);
3. very fine precipitates located inside grains sub-grains (size ranging= <20 nm).

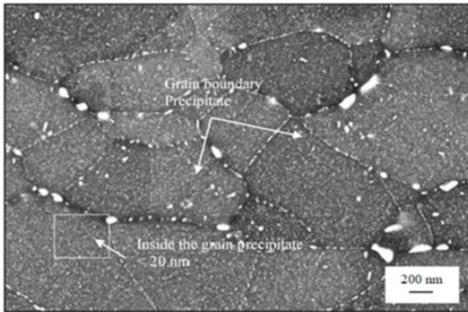


Fig. 4 Sample A microstructure (up-setting T: 293 K)

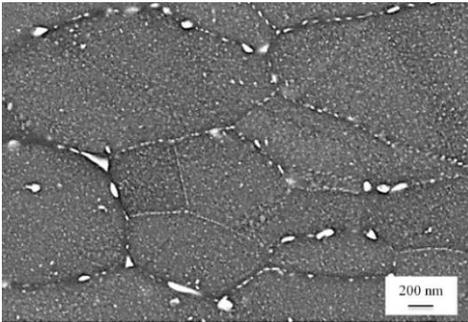


Fig. 5 Sample B microstructure (up-setting T: 423 K)

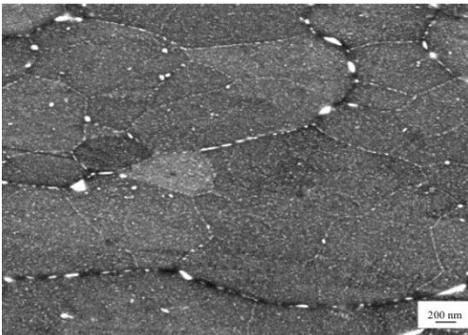


Fig. 6 Sample C microstructure (up-setting T: 473 K)

The dependence of the number of detected precipitates on the upsetting temperature is shown in Figure 7.

In this Figure 7 precipitates are grouped according to their average size, considering the above reported ranges. An increase of the average fine precipitates number with the upsetting deformation temperature is put in evidence in Figure 7. On the other hand it is worth to be noted that the largest precipitates number seems to be almost independent on such process parameter [36-38]. The above statement agrees with the detected improved toughness and yield strength behavior.

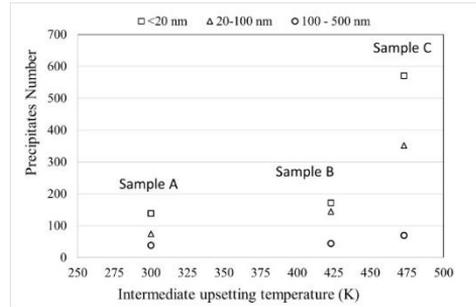


Fig. 7 Mean values of precipitates number grouped in size classes (sample A: intermediate upsetting temperature = 293 K; sample B: intermediate upsetting temperature = 423 K; sample C intermediate upsetting temperature = 473 K)

## CONCLUSIONS

Results of analysis related to AA7050 alloy subjected to warm deformation showed that the precipitation state is very sensitive to intermediate deformation step included in the heat treatment cycle. It is reported that a more evident fine precipitation was found after deformation at 473 K. Such precipitation is claimed to be responsible of a grain refinement effect, hence of toughness  $K_{Ic}$  improvement with respect to materials manufactured according to standard route.

## REFERENCES

1. G. Napoli, A Di Schino, M. Paura, T. Vela: *Metalurgija*, 57, 2018, 111-113.
2. D.K. Sharma, M. Filipponi, A. Di Schino, F. Rossi, J. Castaldi: *Metalurgija*, 58, 2019, 347-351.
3. A.S. Warren: Developments and Challenges for Aluminum – A Boeing Perspective, *Materials Forum*, Eds. J.F. Nie, A.J. Morton, B.C. Muddle, Institute of Materials Engineering Australasia, 28, 2004, 24-31.
4. C. Gashen: *Metals*, 11, 2021, 114. <https://doi.org/10.3390/met11010114>.
5. *Aerospace Structural Materials Handbook*; DoD, Wright-Patterson Air K.F. Force Base: Dayton, OH, USA, 2001.
6. American society of materials, *ASM handbook*, Volume 2, Properties and selection: Nonferrous Alloys and special purpose materials, 2005.
7. O.S. Fayomi, P. Popoola, N. Udoye: in *Aluminium Alloys - Recent Trends in Processing, Characterization, Mechanical Behavior and Applications*, Intech open: Rijeka, 2017. <https://doi.org/10.5772/intechopen.71399>.
8. R.S. Ranam R. Purohit, S. Das: *International Journal of Scientific and Research* 2, 2012, 1-7.
9. D. Manfredi, R. Bidulsky: *Acta Metallurgica Slovaca*, 23, 2017, 276-282. <https://doi.org/10.12776/ams.v23i3.988>.
10. L.F. Mondolfo: *Aluminum Alloys: Structure and Properties*, Butterworth: London, UK, 1976, p. 497-499.
11. X. Sauvage, S. Lee, K. Matsuda, M. Horita: *Journal of Alloys and Compounds*, 710, 2017, 199-202. <https://doi.org/10.1016/j.jallcom.2017.03.250>.
12. J.M. Sanchez, E. Rubio, M. Alvarez, M.A. Sebastian, M. Marcos, J. Mater. Proc. Technol. 164, 2005, 911-918. <https://doi.org/10.1016/j.jmatprotec.2005.02.058>.

13. P. Rambabu, N.E., Prasad, V.V. Kutumbarao, R.J. Wanhill: Aluminium Alloys for Aerospace Applications. In *Aerospace Materials and Material Technologies*; Springer: Singapore, 2017; 1, 9-52.
14. A. Di Schino: *Metals*, 10, 2020, 327.  
<https://doi.org/10.3390/met10030327>.
15. Y.H. Zhao, Z.X. Liao, Z. Hin, R.Z. Valiev: *Acta Materialia*, 52, 2004, 52, 4589-4599.  
<https://doi.org/10.1016/j.actamat.2004.06.017>.
16. K.F. Adam, Z. Long, D.P. Field: *Metallurgical and Materials Transactions A* 48, 2017, 2062-2076.  
<http://dx.doi.org/10.1007/s11661-017-3967-3>.
17. C.G. Parker, D.P. Field: Observation of Structure Evolution during Annealing of 7xxx Series Al Deformed at High Temperature. In *Light Metals 2012*, Springer: Cham, Switzerland, 2012, 383-386.
18. S. Wang, J. Luo, L. Hou, J. Zhang, L. Zhuang: *Materials and Design* 107, 2016, 107, 277-289.  
<https://doi.org/10.1016/j.matdes.2016.06.023>.
19. A. Di Schino, P.E. Di Nunzio: *Acta Metall. Slovaca*, 23, 2017, 62-71,  
<https://doi.org/10.12776/ams.v23i1.852>.
20. D. MacKenzie, D. Scott: *Heat Treating Progress*, 5, 2005, 37-43.
21. AMS 4333 International Standard: *Aluminum Alloy, Die Forgings, 6.2Zn - 2.3Cu - 2.2Mg - 0.12Zr (7050-T7452), Solution Heat Treated, Compression Stress-Relieved, and Overaged*. 2015.
22. R. Wyss, R. Alcoa: *Method for increasing the strength of aluminum alloy products through warm working*, United States Patent: US5194102A, 1993, 16, 03.
23. A. Di Schino, L. Alleva, M. Guagnelli: *Mater. Sci. Forum*, 715-716, 2012, 860-865.  
<https://doi.org/10.4028/www.scientific.net/MSF.715-716.860>.
24. A. Di Schino: *Metalurgija*, 56, 2017, 349-352.
25. S. Gourdet, F. Montheillet: *Acta Materialia*, 50, 2002, 2801-2812. [https://doi.org/10.1016/S1359-6454\(02\)00098-8](https://doi.org/10.1016/S1359-6454(02)00098-8).
26. A. Di Schino, P. Di Nunzio, G.L. Turconi: *Mater. Sci. Forum*, 558-559, 2007, 1435-1441. <https://doi.org/10.4028/0-87849-443-x.1435>.
27. F.J. Humphreys, M. Hatherly: *Recrystallization and related annealing phenomena*, 2nd ed., Elsevier, Amsterdam, 2004.
28. S. Mancini, L. Langellotto, P. Di Nunzio, C. Zitelli, A. Di Schino: *Metals*, 10, 2020, 186.  
<https://doi.org/10.3390/met10020186>.
29. P.J. Jackson: *Mater. Sci. Eng.*, 57(1), 1983, 39-47.  
[https://doi.org/10.1016/0025-5416\(83\)90025-3](https://doi.org/10.1016/0025-5416(83)90025-3).
30. G Saada: *Mater. Sci. Eng. A*, 137, 1991, 177-183.  
[https://doi.org/10.1016/0921-5093\(91\)90333-1](https://doi.org/10.1016/0921-5093(91)90333-1).
31. J.P. Poirier: *Rev. Phys. Appl.*, 11, 1976, 11, 731-739.
32. A.M. Hussein, S.I. Rao, M.D. Uchic, D.M. Dimiduk, J.A. El-Awady: *Acta Mater.*, 85, 2015, 180-190.  
<https://doi.org/10.1016/j.actamat.2014.10.067>.
33. A. Di Schino, C. Testani: *Metals*, 10, 2020 552.  
<https://doi.org/10.3390/met10040552>.
34. C.V. Singh, D.H. Warner: *Acta Materialia*, 58, 2010, 5797. <https://doi.org/10.1016/j.actamat.2010.06.055>.
35. M. van Rooyen, J.A. Sinte Maartensdijk, E. Mittemejer: *Metallurgical Transactions A*, 19, 1988, 2433-2443.  
<https://doi.org/10.1007/bf02645471>.
36. J. Bidulska, R. Bidulsky, M.A. Grande, T. Kvackaj: *Materials*, 12(22), 2019, 3724.  
<https://doi.org/10.3390/ma12223724>.
37. J. Bidulska, R. Bidulsky, T. Kvackaj, M.A. Grande: *High Temperature Materials and Processes*, 27(3), 2008, 203-207.  
<https://doi.org/10.1515/HTMP.2008.27.3.203>.
38. T. Kvackaj, et al.: *Materials Science Forum*, 633-634, 2010, 273-302.  
<https://doi.org/10.4028/www.scientific.net/MSF.633-634.273>.