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A. JARZĘBSKA**, R. BOGUCKI*, M. BIEDA**,#

INFLUENCE OF DEGREE OF DEFORMATION AND AGING TIME ON MECHANICAL PROPERTIES AND MICROSTRUCTURE OF ALUMINIUM ALLOY WITH ZINC

WPŁYW STOPNIA ODKSZTAŁCENIA I CZASU STARZENIA NA WŁAŚCIWOŚI MECHANICZNE I MIKROSTRUKTURĘ STOPU ALUMINIUM Z CYNKIEM.

In order to investigate the influence of the deformation degree and aging time on the mechanical properties and microstructure of AA7050 alloy static tensile test, microhardness measurements, calorimetric analysis and observations of the microstructure in the transmission and scanning electron microscope were carried out. For study a series of cylindrical specimens with an initial diameter of about 3 mm were used. The samples were saturated at a temperature of 470° C for 1 hour and quenched in water. The samples were then subjected to deformation up to the three levels: 0%, 5% and 10%. Deformed samples was artificially aged at 120°C for 6 hours, 12 hours, 24 hours and 72 hours. The results showed that the increase in the degree of deformation caused an increase in yield strength and a decrease in ductility. The longer aging time influenced on an increase in tensile strength, yield stress and microhardness and a decrease in ductility. An analysis of the precipitates present in the material was conducted. The highest value of yield strength equal 538 MPa with elongation 9.2% were obtained for sample pre-strained to 10% and aged for 24 hours. The obtained results showed that prolongation in aging time and use of pre-strain were beneficial for precipitation processes courses, consequently, for optimal mechanical properties of alloy 7050.
Keywords: aluminium alloys, precipitates, tensile strength, TEM

W celu zbadania wpływu stopnia odkształcenia i czasu starzenia na właściwości mechaniczne i mikrostrukturę stopu AA7050 przeprowadzono statyczną próbę rozciągania, badania mikrotwardości, analizę kalorymetryczna oraz obserwacje mikrostruktury w transmisyjnym i skaningowym mikroskopie elektronowym. Do badań wykorzystano serię cylindrycznych próbek o średnicy początkowej równej około 3 mm. Próbki do badań poddano procesowi przesycania w temperaturze 470°C przez 1 godzinę i szybko schłodzono w wodzie. Następnie próbki poddano wstępnemu odkształcaniu o trzy różne stopnie: 0%, 5% i 10%. Później zastosowano proces sztucznego starzenia w temperaturze 120°C w czasie: 6 godzin. 12 godzin. 24 godzin i 72 godzin. Wyniki badań wykazały. że wzrost stopnia odkształcenia powodował wzrost granicy plastyczności i spadek plastyczności. Wydłużenie czasu starzenia wpłynęło na wzrost wytrzymałości na rozciąganie. granicy plastyczności i mikrotwardości oraz na spadek plastyczności. Przeprowadzono analizę występujących w materiale wydzieleń. Najwyższą wartość granicy plastycznej równą 538 MPa przy wydłużeniu 9,2 % osiągnięto dla próbki odkształconej o 10 % i starzonej 24 godziny. Wydłużenie czasu starzenia i stopień odkształcenia wpłynęły korzystnie na poprawę właściwości mechanicznych stopu z gatunku 7050.

1. Introduction

Aluminum alloys of the 7xxx series such as AA7050 are commonly used in the aerospace industry [1-3]. They are used for the production of such parts as fuselage of an aircraft. undercarriage components. The AA7050 alloy is characterized by high mechanical properties and a high ratio of strength to density [4]. The mechanical properties of the alloy are formed by age hardening during which are formed coherent and semi-coherent particles. leading to higher strength. [5-8]. Significant influence on the precipitation processes and increase the strength properties have a plastic deformation after solution heat treatment [9]. Growth the dislocation density facilitates the processes of precipitation and improves the dispersion of particles formed during aging. The advantages influence of microstructure refinement and the processes of severe plastic deformation was reported in several papers [10-12]. In this paper were presented the influence of degree of plastic deformation and aging time on the mechanical properties and microstructure of the alloy AA7050.

2. Experimental procedure

The material used for this work is an AA7050 alloy. The chemical composition of the alloy was investigated on

* CRACOW UNIVERSITY OF TECHNOLOGY, FACULTU OF MECHANICAL ENGINEERING, 24 WARSZAWSKA STR.,31-155 KRAKÓW, POLAND
** INSTITUTE OF METALLURGY AND MATERIALS SCIENCE,POLISH ACADEMY OF SCIENCES, 25 REYMONTA STR., 30-059 KRAKÓW, POLAND
# Corresponding author: m.bieda@imim.pl

spectrometer Bruker Q4TASMAN and the result is shown in Table 1.

TABLE 1

Chemical composition of AA 7050

AA7050	Chemical composition [wt.%]								
	Zn	Mg	Cu	Si	Fe	Cr	Zr	Ti	Al
	5.9	2.0	1.9	0.04	0.23	0.002	0.1	0.04	Bal.

From the rolled thick plate size 6x80x250 mm were cut samples in the longitudinal direction to the rolling direction. Then cylindrical samples were prepared for the tensile test, initial dimension of 3 mm diameter and 25 mm length. The process of preparation material for investigation consists of three stages. In the first step samples was supersaturated at 470°C for 1 hour and then rapidly quenched in water. The temperature of this process was selected on the basis of literature data. The supersaturation time was selected in order to achieve the supersaturation of solid solution and avoid the growth of grain sizes due to the recrystallization [13-15]. The second stage of preparation consisted of plastic deformation after the process of supersaturation. Samples were deformed up to 5 and 10% and compared to samples without deformation. The process of plastic deformation was carried out on a hydraulic testing machine EU 20 to give the elongation 1.25 mm (5%) and 2.5 mm (10%) over a length of 25 mm. The third step comprised the aging which was conducted at a temperature of 120°C [15, 16] for 6 h, 12 h, 24 h, 72 h. Analysis of changes in the mechanical properties of the AA7050 alloy was carried out based on two variables: the aging time and the degree of deformation. Microstructure examinations of the samples were studied using Nikon Eclipse Me 600 optical microscope, Jeol JSM5510LV Scanning Electron Microscope (SEM) and TECNAI F20 Transmission Electron Microscope (TEM). Observations were carried out on longitudinal sections of metallographic specimens etched by "Keler" reagent. The stereological calculations of particles volume fraction were realized using ImageJ software [17]. The analysis of the chemical composition of observed particles were carried out by Joel JSM5510LV Scanning Electron Microscope analyzing of Energy Dispersive Spectroscopy.

Additionally analysis of particles was performed by means of TEM equipped with EDS and HAADF. Electron diffraction registered in TEM was analyzed using KikSPOT software [17]. Differential scanning calorimetry (DSC) was conducted on Netzsch STA409CD thermal analyzer with heating rate of 5°C/min. under the argon gas condition. The microhardness was determined under load of 5N in a Innovatest 400 Series 423 a Vickers microhardness meter. Tensile test was performed in accordance with EN 10002-2:2001 at room temperature on the EU 20 hydraulic testing machine.

3. Results

The microstructural observation of the supply revealed a high degree of processing sheet. Highly elongated grains was observed in both rolling and transverse direction, which are showed in figure 1a,b.

Observation of structure after solution treatment for 1 hour showed a highly elongated grains which proves the absence of recrystallization processes (Fig. 2a). The average value of microhardness was 97 HV. Prolongation of solution treatment time to 2 hours in 470°C caused initiation of recrystallization process and was obtained equiaxial structure (Fig. 2b) with the average value of micro-hardness equal 100 HV.

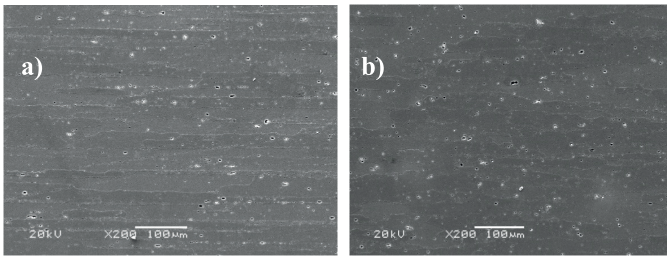


Fig. 1. Microstructure of supply alloy: a) rolling direction. b) transverse direction

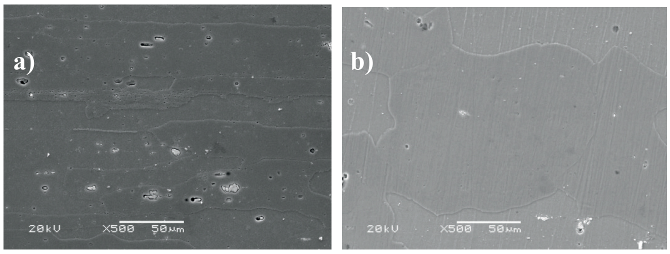
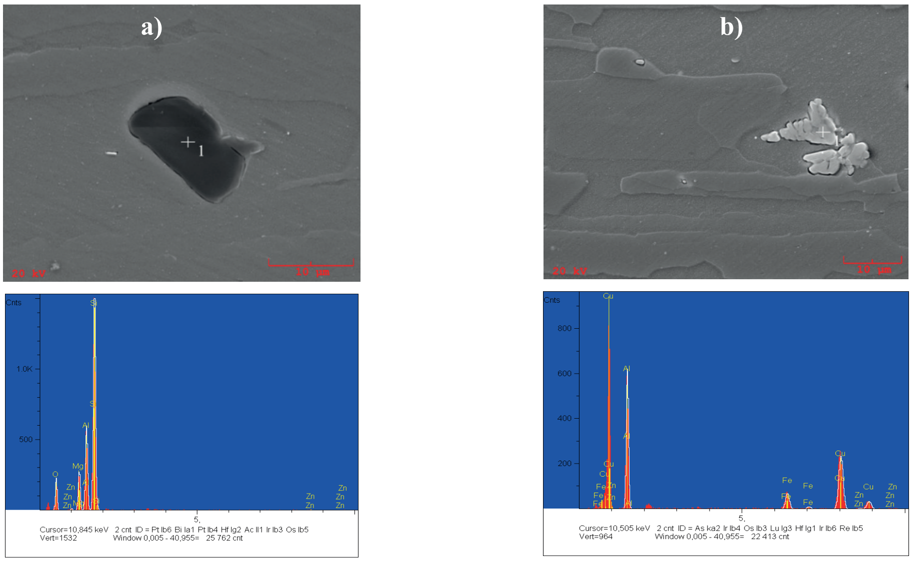


Fig. 2. Microstructure after solution for a period of a) 1 hour – preserved strongly elongated structure. b) 2 hours – recrystallized structure

The microscope observation of non etching samples revealed that in the alloy were primary precipitates [18], which volume fraction Vv was 0.51%. The chemical composition analysis revealed two kinds of primary precipitates. The bright precipitates showed the presence of Al, Cu, Fe, which probably were particles of Al_7Cu_2Fe [19]. The dark precipitates contained in their chemical composition Al, Mg, Si, and low level of Zn and probably were particles of Mg_2Si [20]. Fig 3 shows examples of precipitates and their quantitative and qualitative analysis of chemical composition.

Calorimetric analysis samples were prepared so that the weight was similar to 50 mg. Then heated up at a rate of 5°C/min to temperature 300°C. The result from the trial show fig 4. Based on researches the precipitation processes occurring in the alloy were identified [19]. Five characteristics stages were distinguished. The first one, which occurred in around 50°C, correspond to appearance of GP zones. In the range of the II point (100 - 150°C) GP zones started to dissolve. On the stage III (around 200°C) precipitated phase η'. Phase η' was transformed to phase η on IV stage. Above 250°C, which correspond to stage V, phase η increase, what contributed to decrease their coherence with matrix [20]. The results showed that increase of strain grade caused intensification of precipitation processes.



Elt.	Line	Intensity (c/s)	Atomic [%]	Conc Wt. [%]
O	Ka	51.90	45.6	32.7
Mg	Ka	74.48	6.3	6.9
Al	Ka	168.18	12.4	14.9
Si	Ka	458.92	35.5	44.8
Zn	Ka	1.75	0.2	0.7
			100	100

Elt.	Line	Intensity (c/s)	Atomic [%]	Conc Wt. [%]
Al	Ka	174.63	60.4	39.7
Fe	Ka	34.15	5.2	7.1
Cu	Ka	128.92	33.8	52.2
Zn	Ka	2.01	0.6	1.0
			100	100

Fig. 3. The example of: a) bright precipitate, b) dark precipitate

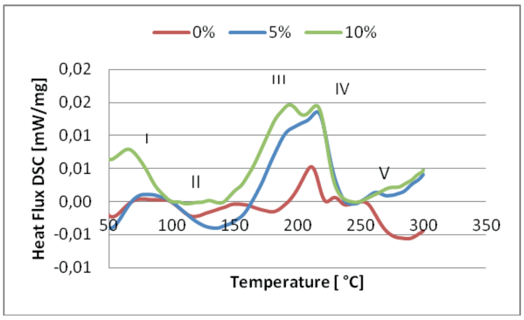


Fig. 4. Calorimetric curves as a function of strain grade

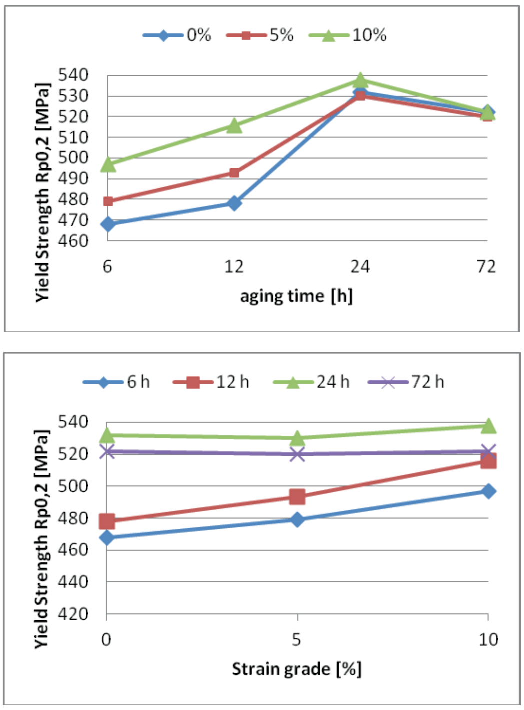


Fig. 5. Change of yield strength as a function of strain grade and aging time

Fig. 5 – 6 show effect of stain grade on mechanical properties during aging. For aging time 6 and 12 hours the yield strength increased steadily with increasing of strain grade (fig.5). For 24 and 72 hours of soaking stabilization of yield strength were observed. The lowest value of yield strength were obtained for sample aged for 6 hours without pre-strain.

For elongation the opposite trend was noticed. For samples aged for 6 and 12 hours elongation decreased. After ageing for 24 and 72 hours the plasticity was on similar level. The highest descent of elongation achieved for sample with 5% of pre-stain and aged for 72 hours. The higher value of yield strength for longer times of aging were caused probably by transformation of GP zones into more stable precipitates η’ and η [14,16,21,22].

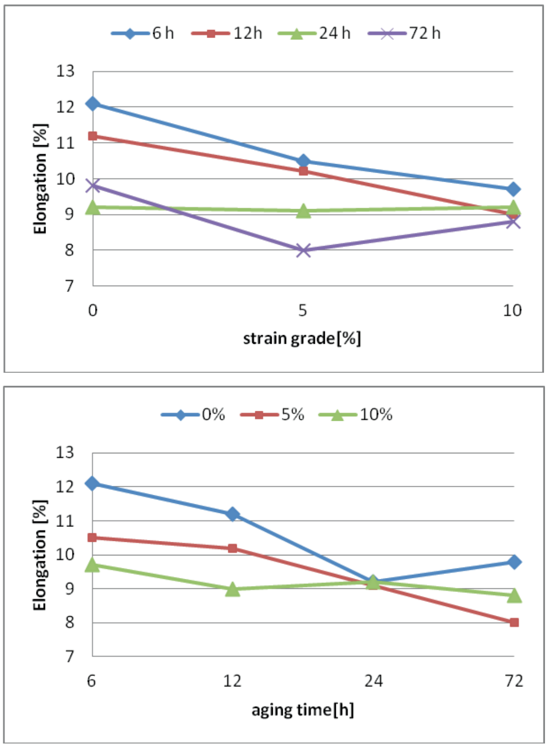


Fig. 6. Change of elongation as a function of strain grade and aging time

TEM investigations performed on samples without deformation (0%) and deformed up to 5 and 10%, and then aged for 24 hours revealed mainly two types of chemical composition of precipitates: with zirconium addition and with zinc and copper addition (Fig. 7). Due to the analysis of selected area diffraction patterns it was possible to distinguish (Fig. 9) the α (Al) and η' phase and metastable Al3Zr $L1_2$ phase[23]. The last two are probably responsible for increase in mechanical properties. Phase Al3Zr can be observed in the fig. 8 as round disperse precipitates of this phase with diameter about 30-50 nm. In the sample pre-strained to 5 % and the aged for 24 h was recognized η phase (Fig. 10) which is probably responsible for small decrease in properties for this sample .

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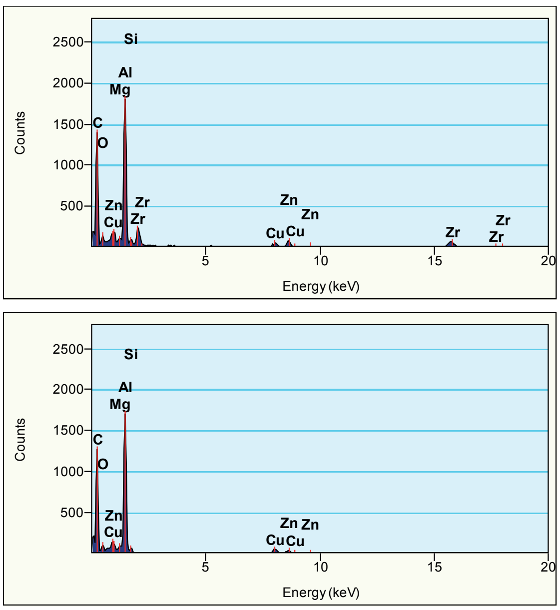

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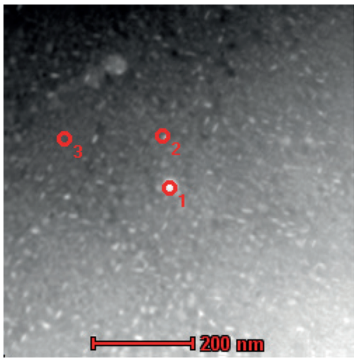

Fig. 7. TEM EDS analysis of particles marked on the STEM HAADF image for 10% pre-strain and 24h of aging

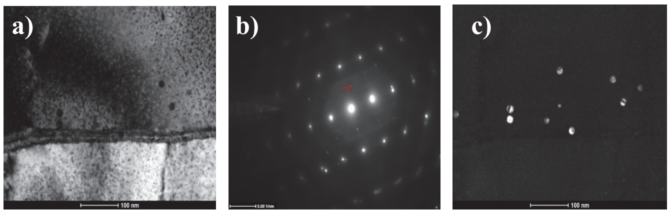

Fig. 8. TEM bright field image a) and corresponding diffraction pattern b) with marked reflex 110 of dark field formation c) for 0% pre-strain and 24h of aging

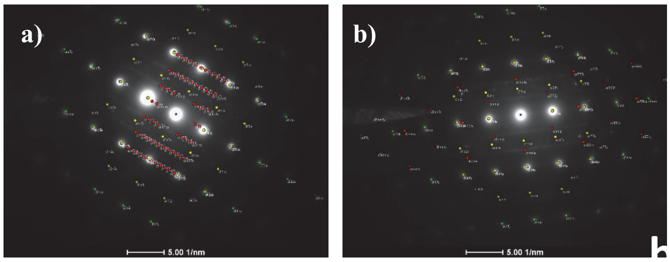

Fig. 9. Selected area diffraction patterns along the [1 -1 2] zone axes of Al matrix with marked α (Al) (green) and η' phase(red) and metastable i Al3Zr L12 (yellow) for a) 5% pre-strain and 24 h aging b) and 10% pre-strain and 24 h aging

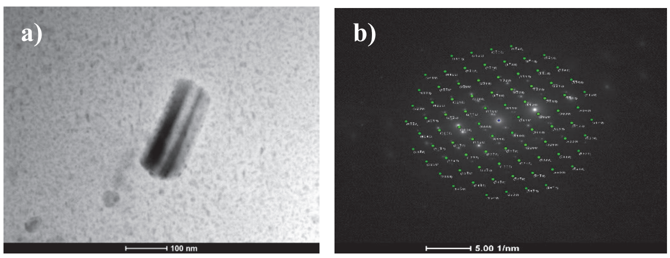

Fig. 10. Bright field image a) and selected area diffraction pattern b) with marked η phase for 5% pre-strain and 24 h aging

Both strain grade and aging time had influence on mechanical properties. Grow of yield strength were observed up to 24 hours. Prolongation time of aging to 72 hours weren't caused increase of endurance. In case of extension of aging time was observed a decrease of plasticity.

4. Discussion

There was observed that strain grade and aging time had influence on the level of obtained mechanical properties. The changes were due to precipitation processes of secondary phases and their transformation into more stable phases.

After solution for one hour the extended structure was preserved, which proves the delayed of recrystallization processes.

Decrease of yield strength and tensile strength and increase of ductility after solution treatment is correspondent to dissolved of GP zones and precipitation of phases η' and η. The processes of recovery also could provoked these changes.

Significant effect of strain grade on course of particular processes was observed. For samples with 10% of pre-strain were detected the most intensive processes of precipitation. 10% of pre-strain provoked faster start of precipitation in lower temperature, what should explain higher amount of dislocations. This involves a higher amount of privileged places for nucleation of secondary phases. Increase of yield strength in pre-strained samples with aging time 6 and 12 hours was caused presumably by higher density of dislocations, so that nucleation of GP zones were more intense. Extension of aging time weren't caused significant differences in the level of yield strength. It can be explained by the softening of matrix due to mobilization of structural renewal processes. As well as dissolve of GP zones and nucleation of η' and η phases cannot be ignored.

The opposite trend was observed for elongation. Prolongation of aging time provoked decrease of plasticity. The results of microhardness and tensile strength as a function of aging time had similar course. In case of undeformed and pre-strained to value of 5% samples were noticed growth of micro-hardness and tensile strength for all aging times. For samples pre-strained to 10% increase of durability were observed only to 24 hours. Decrease of tensile strength after aging for 72 hours should be connected with overaging and structural renewal.

Prolongation of aging time in connection with increase of strain grade leads to intensification on nucleation process as well as accelerate the transformation of secondary phase into more stable. This is confirmed by calorimetric analysis results, which revealed increase intensity of nucleation processes and movement to the lower temperature with increasing of pre-strain.

The highest value of yield strength equal 538 MPa with elongation 9.2% were obtained for sample pre-strained to 10% and aged for 24 hours. This was a strength increase by more than 100 MPa, compared to the initial condition (initial condition yield strength = 437 MPa, elongation = 8.3%). The highest elongation equal 12.1% was obtained after 6 aging hours for undeformed sample which was also better in initial condition.

The obtained results showed that use of pre-strain were beneficial for precipitation processes courses. consequently. for optimal mechanical properties.

5. Conclusions

1. After solution in 470°C for 1 hour in researched alloy AA7050 the recrystallization process wasn't observed. It was probably caused by presence of Al3Zr metastable phase which delayed recrystallization process.
2. Increase of strain grade to 10% had a good influence on mechanical properties of AA7050.
3. Strain grade affected on intensity of precipitation processes. For sample pre-strained to 10% was observed movement of nucleation process initiation to lower temperatures.
4. Prolongation of aging time above 24 hours had no influence on increase of durability of AA7050.
5. The highest value of yield strength was obtained for pre-strained to 10% and aged for 24 hours sample, which amounted 538 MPa with elongation 9.2 %.
6. Decrease of mechanical properties which was observed after 5% of pre-strained with longer times of aging above 6 hours were probably provided by effect of dissolve GP zones and transformation of η' into η phases.

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REFERENCES

[1] T. Warner, Mater. Sci. Forum **519-521**, 1271 (2006).
[2] J. Liu, Mater. Sci. Forum **519-521**, 1233 (2006).
[3] N. Li, X. Lu, J. Cui, T. Nonferr. Metal Soc. **18**, 541 (2008).
[4] K. Stiller, P.J. Warren, V. Hansen, J. Angenete, J. Gjonnes, Mater. Sci. Eng. **270(1)**, 55 (1999).
[5] B. Cai, B.L. Adams, T.W. Nelson, Acta Mater. **55**, 1543 (2007).
[6] J.D. Robson, P.B. Prangnell, Mater. Sci. Technol. **18(6)**, 607 (2002).
[7] A.J. Wert, N.E. Paton, C.H. Hamilton, W.M. Mahoney, Metall. Transaction. **12A**, 1267 (1981).
[8] Y. Lang, H. Cui, Y. Cai J. Zhang, Mater. Des. **39**, 220 (2012).
[9] Z. Li, B. Xiong, Y. Zhang, B. Zhu, F. Wang, H. Liu, Mater. Process. Tech. **209**, 2021 (2009).
[10] M. Furukawa, Z. Horita, M. Nemoto, R.Z. Valiev, T.G. Langdon, Acta Mater. **44(11)**, 4619 (1996).
[11] R.Z. Valiev, N.A. Enikeev, M. Murashkin, V. Kazykhanov, X. Sauvage, Scripta Mater. **63**, 949 (2010).
[12] K.R. Cardoso, D.N. Travessa, W.J. Botta, J. Jorge, Mater. Sci. Eng. A **528**, 5804 (2011).
[13] K. Shen, J. Chen, Z. Yin, T. Nonferr. Metal Soc. **19(6)**, 1405 (2009).
[14] J. Zang, K. Zhang, S. Dai, T. Nonferr. Metal. Soc. **22**, 2638 (2012).
[15] P. Li, B. Xiong, Y. Zhang, Z.T. Li, Nonferr. Metal. Soc. **22**, 546 (2012)
[16] X.W. Li, B.Q. Xiong, Y.A. Zhang, C. Hua, F. Wang, B.H. Zhu, H.W. Liu, Sci. China Ser. E **52**, 67 (2009)
[17] KIKSPOT software – www.crystorient.com
[18] Y.C. Lin, Y.Q. Jiang, X.M. Chen, D.X. Wen, H.M. Zhou, Mater. Sci. Eng **588**, 347 (2013).
[19] S.A. Barter, N. Athiniots, L. Lambrianidis, Aircraft Materials Technical Memorandum 1990.
[20] D. Wang, D.R. Ni, Z.Y. Ma, Mater. Sci. Eng. **494**, 360 (2008).
[21] G. Sha, A. Cerezo, Surf. Interface Anal. **36**, 564 (2004).
[22] G. Sha, A. Cerezo, Acta Mater. **52**, 4503 (2004).
[23] Litynska-Dobrzynska L Arch. of Met. And Mater. **51**, 555 (2006).