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IMPROVEMENT OF MECHANICAL PROPERTIES OF A NANOALUMINIUM ALLOY BY PRECIPITATE STRENGTHENING

POPRAWA WŁAŚCIWOŚCI MECHANICZNYCH NANOKRYSTALICZNEGO STOPU ALUMINIUM POPRZEZ UMOCNIENIE WYDZIELENIOWE

In the present study, severe plastic deformation (SPD) processing was combined with pre- and post processing heat treatment to investigate the possibility of synergic grain size and precipitation strengthening. Samples of 7475 alloy were solution heat treated and water quenched prior to hydrostatic extrusion (HE) which resulted in a grain refinement by 3 orders of magnitude, from 70 μm to about 70 nm. The extruded samples were subsequently aged at temperatures resulting in formation of nanoprecipitates.

Keywords: aluminium alloys, nanostructured metals, severe plastic deformation (SPD), hydrostatic extrusion (HE), precipitate strengthening, mechanical properties

W pracy zbadano możliwość zwiększania wytrzymałości stopów aluminium poprzez połączenie zabiegów rozdrobnienia ziaren z wykorzystaniem metod SPD z obróbką cieplną. Próbki stopu aluminium 7475 poddano wciskaniu hydrostatycznemu w stanie przesyconym, co spowodowało znaczące rozdrobnienia ziarna, z 70 μm do 70 nm. Wyciśnięte próbki poddano starzeniu w celu wytworzenia nanowydzieleń.

1. Introduction

The 7xxx series aluminium alloys (Al-Zn-Mg-Cu) are widely used in aerospace industry because of their high strength (about 500 MPa). In conventional micrograined alloys, such properties can be obtained via precipitation hardening, efficiency of which depends on precipitates size and number density [1-3]. One of the strengthening mechanisms which can also be considered in the context of these alloys is grain size refinement, possibly down to the nanoscale level. Such nanograined structures can be produced, among other methods, by SPD [4-7]. Extensive research programme conducted at Warsaw University of Technology and Institute of High Pressure Physics has shown that hydrostatic extrusion (HE) is an efficient SPD method for production of many nanostructured metals and alloys in a variety of forms (rods, wires, tubes) [8-12]. However, a question arises whether or not nanostructured alloys can additionally be strengthened by precipitation.

The combination of precipitation strengthening and grain size refinement down to nanoscale was applied to various aluminium alloys [13-15]. However, the aging of nanostructured aluminium alloy has not been fully explored yet. In our previous work [16], it was shown that in a case of nanograined 7475 alloy conventional aging temperatures do not improve mechanical properties. In the present work, we report original results providing more details on the nanoprecipitation in nanograined 7475 aluminium alloy.

2. Experimental

The material used in this study was an Al-Zn-Mg-Cu 7475 alloy with chemical composition given in Table 1. The solution heat treated and water quenched samples were processed by HE at room temperature in three passes with a total true strain of about 4. The extruded specimens were water cooled at the die exit and subsequently aged at three temperatures: 100, 130 and 160°C for various aging time. Solution heat treated and water quenched micrograined samples were used as a reference material.

The specimens for tensile tests, cut parallel to extrusion direction and machined using spark erosion, had

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a cross section of 0.6×0.8 mm and a gauge length of 5 mm, as illustrated in Fig. 1. The tensile tests were performed at room temperature at a strain rate of 10^{-3} s^{-1}.

TABLE 1
Chemical composition of 7475 aluminium alloy (wt%)

Alloy	*Zn*	*Mg*	*Cu*	*Zr*	*Fe*	*Si*	*Ti*	*Mn*
7475	6.00	2.49	1.66	0.12	0.12	0.094	0.015	0.01

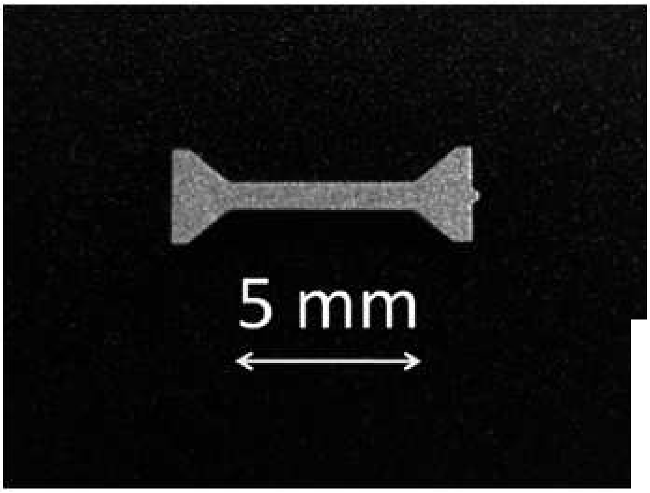

Fig. 1. Sample used in minitensile test

The microstructures of aged samples were invesı gated using a high resolution scanning transmission ele tron microscope Hitachi HD 2700. Thin foils were c perpendicularly to the extrusion direction.

3. Result and discussion

Microstructure observations revealed a coar grained microstructure with an average grain diamet of ~70 μm in the samples after pre-processing treatment (solution annealing and water quenching). HE processing resulted in a significant reduction of the grain size down to 66 nm. This microstructural transformation brought about a significant increase in mechanical strength, i.e. the yield strength (YS) increased from 205 to 650 MPa whereas ultimate tensile strength (UTS) from 360 to 700 MPa, as in details described in [17].

The results given in Table 2 and Fig. 2 illustrate changes in mechanical properties caused by post-HE aging at various temperatures. It should be pointed out that for post-HE aging temperatures of 130 and 160°C, which are recommended for 7475 aluminium alloy [18], both YS and UTS decrease. Only relatively low aging temperature at 100°C for long aging times brings about an increase in strength. This result indicates that effective aging conditions for nanograined alloys are shifted to lower temperatures when compared to conventional microcrystalline ones.

TABLE 2
The influence of the aging time and temperature on mechanical properties of the nanostructured aluminium alloy

time [h]	**temperature [°C]**	**YS [MPa]**	**UTS [MPa]**	**Elongation [%]**
2	100	598	645	2,2
	130	623	644	1,2
	160	569	600	3,8
24	100	656	676	1,4
	130	554	620	4,0
	160	453	497	6,1
after HE		619	679	2,4

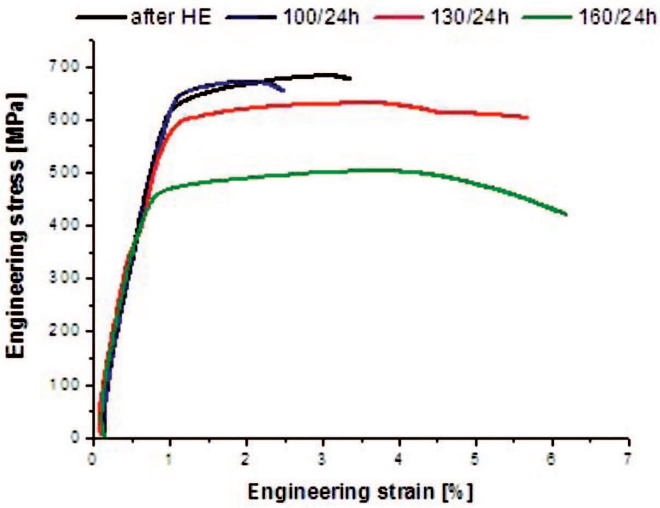

Fig. 2. The comparison of the tensile curves of a nanograined aluminium alloy aged at 100, 130 and 160°C for 24 hours

The mechanical properties of micro- and nanograined samples aged at 100°C for various times are presented in Table 3 and in Fig. 3. From these results, one can conclude that the combination of SPD processing and low temperature (100°C) aging leads to a significant improvement in the mechanical properties. However, it should be noted that precipitation strengthening in the case of nanograined samples is not as effective as for micrograined ones. In the case of micrograined samples, aging increases YS by 48%, whereas in the case of nanograined ones only by ~12%. Also, the simultaneous improvement of both mechanical strength and plasticity should be pointed out for the nanograined sample aged at 100°C for 54 hours.

TABLE 3

The comparison of the mechanical properties for micro- and nanograined aluminium alloy after aging at 100°C with different time

time[h]	YS [MPa]		UTS [MPa]		Elongation [%]	
	micro	nano	micro	nano	micro	nano
2	364	598	520	645	21,6	2,2
24	527	615	580	676	12,9	1,4
54	540	**688**	619	**705**	12,5	2,7
after HE		**619**		**679**		2,4

In order to explain the measured mechanical properties, HRSTEM (high resolution scanning transmission electron microscope) investigations have been performed. These investigations revealed that the aging of both nano- and micrograined aluminium alloy leads to the formation of nanoprecipitates uniformly distributed in the entire volume of the material (Fig. 4). One can observed that with increasing aging time the precipitation size also increases, in both nano- and micrograined samples. It should also be pointed out that precipitates in nanoaluminium are significantly smaller than those formed in micrograined samples after the same annealing conditions.

In nanograined samples, one can also see relatively large number of precipitates at grain boundaries, which are very likely formed during HE deformation, as they are also present in the microstructure immediately after HE [19], and grow during subsequent aging. Obviously these precipitates do not contribute to the precipitation strengthening in the same way as those in the grain interiors. On the other hand, they may improve thermal stability, particularly at the lower aging temperatures [19], as the nanograined samples aged at 100°C for 54 hours maintain grain diameter of 87 nm. The precipitates located at grain boundaries are probably the equilibrium η phase. Inside the grains, one can see a mixture of GP zones and metastable η' precipitates, which mainly contribute to the strengthening of the material.

Less efficient precipitation strengthening of nanograined sample can be thus explained by enhanced precipitation at grain boundaries which constitute a major microstructural elements. Simple calculation shows that the surface area of grain boundaries in nanograined sample is higher by 3 orders of magnitude than compared to micrograined one.

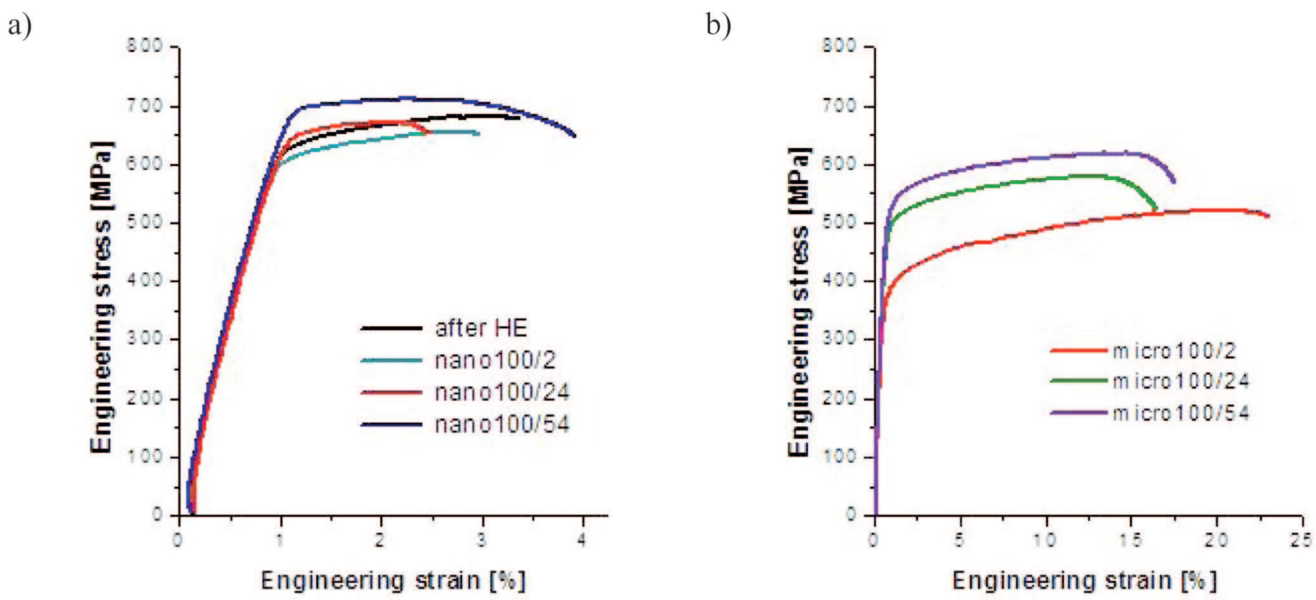

Fig. 3. The comparison of the tensile curves of a nano- (a) and micrograined (b) aluminium alloy aged at 100°C

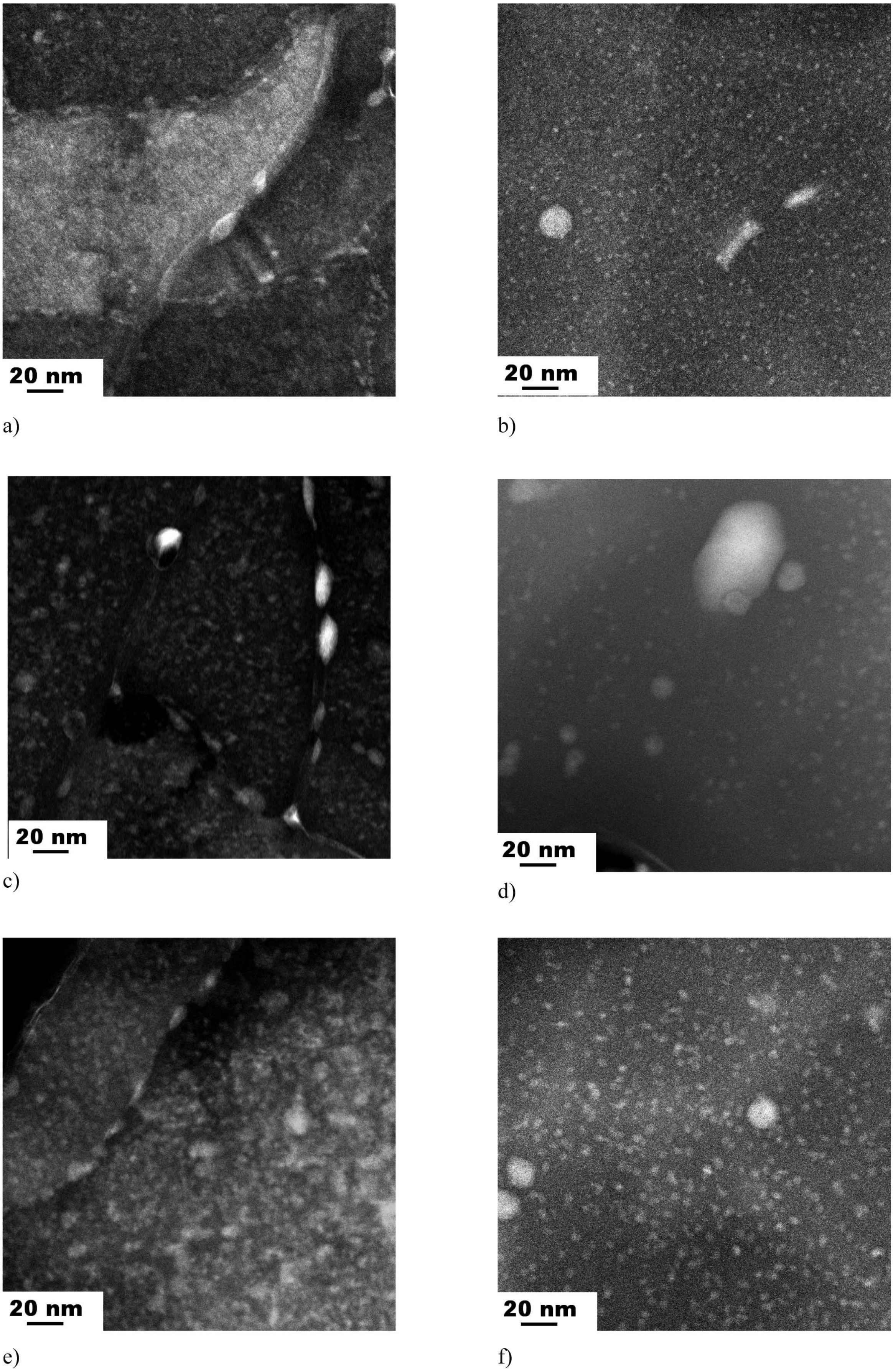

Fig. 4. The images of microstructure in the nano- (a, c, e) and micrograined (b, d, f) sample after aging at 100°C for: 2, 24 and 54 hours, respectively

4. Summary

The results obtained show superior properties of Al7475 alloy after HE-processing of solution treated billets. It has been also demonstrated that further increase in the strength of this alloy can be obtained by post-HE aging. However, aging conditions for nanograined alloys are shifted to lower temperatures because of an enhanced diffusion rate due to a high density of defects accumulated during SPD processing. The results also suggest that the precipitation strengthening in nanograined alloy is not as effective as in micrograined ones mainly due to enhanced precipitation at grain boundaries.

Further research in progress is focused on the size, shape and spatial locations of precipitates in nanograined samples with the aim of optimizing the heat treatment of HE-processed Al7475 alloy.

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