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Effect of annealing and additional deformation on the dynamic properties of ultrafine-grained AMg4.5 alloy

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The possibility of increasing the static and dynamic strength of the aluminum-magnesium alloy AMg4.5 is shown by using methods of severe plastic deformation, a combination of annealing and additional deformation of ultrafine grained alloy. The strength properties of the material are evaluated using a structural-temporal approach to the analysis of experimental data on the dynamic tension of small samples obtained on split-Hopkinson pressure bar.

Keywords: dynamic strength, Incubation time criterion, Kolsky method, aluminum.

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Introduction

Application of aluminum alloys of the Al-Mg system in industry, including critically important units, is due to increased strength and performance characteristics. The alloys of this series are strain-hardenable [1]. The increased percentage of magnesium content increases the strength in the static and dynamic range of loading with a slight decrease in plastic characteristics [2]. At the same time, the strength characteristics increasing of alloys of Al-Mg system by standard methods is limited. Example of the AMg4.5 alloy [3] shows the possibility of increasing the material strength in the static loading range by three using the methods of severe plastic deformation (SPD) was shown [4-10]. In dynamic tension experiments the aluminum alloy in the ultra-fine grain (UFG) state was destroyed at stresses lower than its static strength. First of all, the obtained results were associated with significant decrease in the plastic characteristics of the material. Also, the behavior of the material in the UFG state in the dynamic range was not sufficiently studied and, despite the fact that, as a rule, the ultimate strength of the material increases with increase in the strain rate, reverse regularities can be observed, they are associated with the structural and temporal features of the destruction process [11–15].

In this paper we discussed the possibility of increasing the static and dynamic strength of the AMg4.5 alloy using a combination of methods of severe plastic deformation, annealing, and additional deformation of the alloy in the UFG state.

1. Studied materials and experimental techniques

Aluminum alloy AMg4.5 (Al-4.56Mg-0.46Mn-0.32Fe-0.21Si (wt.%)) was in the as-cast state. The structure was modified by high pressure torsion (HPT) on Walter-Klement GmbH press. Primary HPT treatment for 10 turns (n = 10) was carried out at room temperature under a pressure of 6 GPa. As a result of deformation, disks 20 mm in diameter and 1.6 mm thick were formed. The true logarithmic degree of material deformation at the middle of the disk radius was $e \approx 5.5$ [16].

Additional modes of deformation-heat treatment included annealing at temperatures of $100-450^{\circ}$ C for 1 h. Some of the samples were subjected to additional HPT treatment by 0.25 turn at pressure of 6 GPa, which corresponds to a true deformation of $e \approx 1.5$.

The microstructure of the material in various states was studied by transmission electron microscopy (TEM) and X-ray diffraction analysis (XDA). The TEM studies were carried out on a Zeiss Libra 200FE microscope; XDA was performed on a Bruker D8 DISCOVER diffractometer. Full-profile modeling by the Pauli method using "TOPAS 5.0" software determined the average size of coherent scattering regions (*C*) and the level of crystal lattice microdistortions $(\langle \varepsilon^2 \rangle^{1/2})$. The dislocation density was determined by the formula [17]:

$$L_{dis} = \frac{2\sqrt{3}\langle \varepsilon^2 \rangle^{1/2}}{C_b}$$

where b – is the Burgers vector of the dislocation.

According to the results of mechanical tests in the quasistatic range of loads, the average values of conditional yield stress ($\sigma_{0.2}$) corresponding to 0.2% of deformation, ultimate

Samples	HV, MPa	$\sigma_{0.2}$, MPa	σ_{UTS} , MPa	$ au$, μ s	$\delta, \%$	$\delta_1,\%$
AMg4.5 HPT10	$\begin{array}{c} 780\pm3\\2180\pm8\end{array}$	$\begin{array}{c} 120\pm2\\ 725\pm1\end{array}$	$\begin{array}{c} 240\pm 5\\ 725\pm 2\end{array}$	12	11 0	8.8 0
HPT10_200C HPT10_200C + 0.25	$\begin{array}{c} 1990 \pm 15 \\ 1950 \pm 14 \end{array}$	$\begin{array}{c} 518\pm2\\ 329\pm1\end{array}$	$\begin{array}{c} 518\pm5\\ 653\pm3\end{array}$	3.4 2.7	0 2	0 2

Table 1. Mechanical properties of AMg4.5 alloy samples

strength (σ_{UTS}), ultimate elongation (δ), relative elongation to failure (δ) and relative uniform elongation (δ_1) were determined.

Dynamic tension experiments were carried out according to the Kolsky method using split Hopkinson rods [18,19] on samples similar to those used under quasi-static loading conditions, with a work part 5 mm long and 2 mm wide. The unit consists of a pneumatic loading device with a caliber with the gage of 30 mm, striker 400 mm long, split Hopkinson pressure bars with the diameter of 20 mm and loading bar length of 3000 mm, as well as a measuring bar $-1500 \,\mathrm{mm}$ [3]. The striker is accelerated by compressed air supplied by the compressor into the chamber, pressure is monitored by a pressure gage. Variation of the impactor speed is carried out by changing the pressure from 3 to 8 bar. The threshold quantity to characterize the sample failure under impact loading in this paper is the dependence of the maximum breaking stress on stress increase rate. The experimental points were used to determine the parameter τ , which is responsible for the dynamic strength of the material according to the structuraltemporal approach.

The fractography of the sample surface destroyed under uniaxial tension was performed by scanning electron microscopy (SEM) on a Zeiss AURIGA Laser microscope.

2. Experimental results and discussion

The tensile strength of the material after HPT treatment for 10 turns increased relative to the strength of the initial state from 240 to 725 MPa. The offset yield strength increased from 120 to 725 MPa, the ultimate elongation decreased from 11% to values close to 0%. The high strength of the material and the inability to plastically deform led to decrease in the material strength with increase in the rate of stress growth [3]. In order to increase the material plasticity, a set of heat-strain treatments of the material in the UFG state was carried out. The optimal characteristics for the conditions of quasi-static and dynamic loading were obtained by additional annealing of the material at a temperature of 200°C for 1 h and subsequent HPT treatment by 0.25 turn at pressure of 6 GPa.

Table 1 presents the mechanical characteristics of the AMg4.5 alloy in various structural states. It can be seen that the additional annealing of the alloy with the UFG structure at 200° C reduces the material strength, but does not add plastic properties. The subsequent HPT treatment by 0.25

 Table 2. Structural analysis results

State	d, nm	C, nm	$\langle \varepsilon^2 \rangle^{1/2}$	L_{dis}, m^{-2}
AMg4.5	41 000		0.00016	_
HPT10	108	20	0.00084	9.4 · 1013
HPT10_200C	191	203.235	0.00018	$1.1\cdot10^{13}$
HPT10_200C + 0.25	135	139	0.0007	$6.4\cdot10^{13}$



Figure 1. Experimental and calculated dependences of maximum tensile strength on the stress growth rate of the AMg4.5 alloy in the initial CG and UFG states (markers — experimental data, the curves are plotted according to the dependence (1) taking into account the material parameters from Table 1).

turn under pressure of 6 GPa decreases the yield strength to 329 MPa, increases the strength to 653 MPa at a plastic deformation level of 2%.

The results obtained can be partly explained by the change in the structure parameters presented in Table 2. The initial HPT treatment by 10 turns (HPT10) leads to a significant grain size refinement (d), which, in combination with a high level of microstresses, negatively affects the plasticity of the material.

During heat treatment of the material with UFG structure (HPT10_200C), the level of microstresses decreases, but at the same time the dislocation density decreases, which mostly determines the plasticity and strength of the material. Additional HPT treatment by 0.25 turns even more makes it possible to reduce the level of microstresses and to increase the density of dislocations. The average grain



Figure 2. SEM data. Failure surface of the AMg4.5 alloy in the initial CG state under dynamic tension.



Figure 3. SEM data. Destruction surface of samples from HPT10 under dynamic tension conditions.

size in this state corresponds to the size characteristic for the material after the initial HPT treatment, which explains the high strength of the material (HPT10_200C + 0.25) by Hall–Petch law [20,21].

To check the characteristics of new materials under impact loads the dynamic tension experiments were carried out using a setup that implements the scheme of split Hopkinson rods. It was shown that the material in the UFG state is capable for dynamic hardening. Fig. 1 shows experimental dependences of the maximum tensile strength on the stress growth rate of the AMg4.5 alloy in the initial coarse-grain (CG) and UFG states.

To analyze the strength characteristics of the material under conditions of quasi-static and dynamic loading the incubation time criterion was applied [22]:

$$\frac{1}{\tau} \int_{t-\tau}^{t} \frac{\sigma(s)}{\sigma_{UTS}} ds \le 1, \tag{1}$$

where t — time, σ — breaking stress vs. time, σ_{UTS} — ultimate tensile strength under quasi-static loading, τ —

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incubation time of failure accountable for material dynamic strength. The threshold value of the material strength for each value of the stress growth rate is determined at the moment of reaching equality in equation (1). The parameter τ is calculated using the least squares method (LSM) as the optimal value that minimizes the standard deviation of the calculated values of the material strength from the experimental points. In Fig. 1, for each state of the material the calculated curves are plotted according to the dependence (1), taking into account the parameters of the material from Table 1, illustrating the maximum tensile strength of the material depending on the change of stress growth rate.

The aluminum alloy in the initial state has the highest dynamic strength in terms of incubation time. Fig. 2, a shows the general plan of the destruction surface of the sample under impact loading. It can be seen in Fig. 2, b that the destruction surface is formed by micropores with an heterogeneous distribution of shape and size. In some areas on the destruction surface, one can note the presence of microcracking areas marked with a frame. This indicates the activation of a limited number of micropore nucleation



Figure 4. SEM data. Destruction surface of samples HPT10_200C under dynamic tension conditions.



Figure 5. SEM data. Destruction surface of samples HPT10_200C + 0.25 under conditions of dynamic tension.

sites. Meanwhile, the areas of plastic deformation make up a large part of the failure surface. Returning to the data analysis presented in Fig. 1, note that the revealed features of material destruction in the region of dynamic deformation lead to limitation of the threshold values of the growth rate of stress and strength, which is significantly lower than the response of materials in UFG states. The inertia of the material is limited by the heterogeneous formation and distribution of plasticity zones, which leads to the samples destruction at stresses not exceeding 350 MPa.

The destruction surface of the AMg4.5 alloy after HPT treatment by 10 turns under dynamic tension is shown in general plan in Fig. 3, *a*. Destruction can be characterized as brittle intergranular, since the pits on the sample surface have a uniform pattern with characteristic sizes of 100-300 nm (Fig. 3, *b*), which corresponds to the grain size in the material after HPT treatment by 10 turns. Also note that during the impact wave passage in local areas the extreme values of forces can be formed, which, in the absence of the possibility to implement plastic deformation

mechanisms, leads to the samples destruction in the event of increase in the strain rate at stress values significantly below the threshold values obtained under quasi-static loading conditions.

Additional heat treatment of the material with UFG structure at 200°C for 1 h leads to decrease in the material strength to 520 MPa in the quasi-static range of loads and change in the nature of destruction under impact loading. Fig. 4, *a* shows the destruction surface of the sample under impact loading. In Fig. 4, *b*, with magnification, one can see the pitted nature of the destruction with local areas of brittle chipping.

Additional HPT treatment of the material with UFG structure led to increase in the static strength of the material up to 653 MPa and the appearance of plasticity, which is a necessary condition for the material deformation under conditions of high-speed impact loads. The destruction surface is shown in general plan in Fig. 5, a. It can be seen in Fig. 5, b that the surface is formed by uniform pits with local combination of pores, in the depth of which areas of

brittle chipping are visible. The combination of high strength and ductility in the material makes it possible, in the case of impact loading, to achieve strength values of 850 MPa, which is by 30% higher than the extreme values obtained under quasi-static deformation conditions.

Analyzing data presented in Fig. 1 and the dynamic strength of materials in terms of incubation time, we can conclude that despite a significant change in the structure and properties of the AMg4.5 aluminum alloy during deformation treatment, its behavior under impact loading conditions is predictable. The response of the material with UFG structure to impact action can be described by a calculated curve plotted according to the criterion of incubation time, taking into account the parameters of the material with CG structure ($\sigma_{UTS} = 240$ MPa, $\tau = 12 \,\mu$ s).

Conclusion

HPT treatment of the strain-hardenable AMg4.5 aluminum alloy made it possible to significantly increase the ultimate strength of the material (by \sim 3 times), while there was a complete loss of plasticity and dynamic strength of the material. The plasticity increasing (up to $\sim 2\%$) while maintaining a high tensile strength was obtained by additional strain-heat treatment, including annealing at 200°C for 1 h and additional torsion deformation by 0.25 turns. It is shown that the simultaneous achievement of strength and plasticity is obtained by introducing additional dislocation density into the structure.

In experiments on dynamic tension using a setup that implements the Kolsky method, the material modified in this way showed the effect of hardening with increasing strain rate. Analysis of the fractography of the destroyed samples surface did not reveal obvious signs of intergranular destruction. It is most likely that the dislocation density introduced into the structure was mostly concentrated at the grain boundaries, which made it possible to implement the mechanisms of plastic deformation under impact loading conditions.

Using the incubation time criterion for experimental data in the area of quasi-static and dynamic loading of the alloy with CG and UFG structures, the material parameters were determined and the calculated dependences of the maximum tensile strength on the stress growth rate were plotted. A good agreement between the calculated curves and the experimental points is obtained over the entire range of parameters changes of the external loading.

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Conflict of interest

The authors declare that they have no conflict of interest.

References

- [1] L.F. Mondolfo. *Aluminum Alloys: Structure and Properties* (Elsevier, 2013)
- [2] A.D. Evstifeev, G.A. Volkov. Procedia Structural Integrity, 28, 2261 (2020). DOI: 10.1016/j.prostr.2020.11.059
- [3] A.D. Evstifeev. ZhTF, 92 (9), 1349 (2022). (in Russian) DOI: 10.21883/JTF.2022.09.52926.33-22
- [4] Y. Zhu, R.Z. Valiev, T.G. Langdon, N. Tsuji, K. Lu. MRS Bull., 35, 97 (2010). DOI: 10.1557/mrs2010.702
- [5] Y. Estrin, A. Vinogradov. Acta Mater, 61 (3), 782 (2013).
 DOI: 10.1016/j.actamat.2012.10.038
- [6] T.G. Langdon. Acta Mater., 61 (19), 7035 (2013).
 DOI: 10.1016/j.actamat.2013.08.018
- [7] I. Sabirov, M.Yu. Murashkin, R.Z. Valiev. Mater. Sci. Eng. A, 560, 1 (2013). DOI: 10.1016/j.msea.2012.09.020
- [8] M. Kawasaki, T.G. Langdon. J. Mater. Sci., 49, 6487 (2014).
 DOI: 10.1007/s10853-014-8204-5
- [9] A.M. Mavlyutov, T.A. Latynina, M.Yu. Murashkin, R.Z. Valiev, T.S. Orlova. FTT, **59** (10), 1949 (2017). (in Russian).
 DOI: 10.21883/FTT.2017.10.44964.094 [A.M. Mavlyutov, T.A. Latynina, M.Yu. Murashkin, R.Z. Valiev, T.S. Orlova. Physics Solid State, **59** (10), 1970 (2017).
 DOI: 10.1134/S1063783417100274]
- [10] R.Z. Valiev, I.V. Alexandrov, Y.T. Zhu, T.C. Lowe. J. Mater. Res., 17, 5 (2002). DOI: 10.1557/JMR.2002.0002
- G.V. Stepanov, V.V. Astanin, V.I. Romanchenko, A.P. Vashchenko, V.M. Tokarev, B.D. Chukhin, Y.P. Guk. Strength Mater., 15, 220 (1983).
 DOI: 10.1007/BF01523474
- [12] A.A. Gruzdkov, E.V. Sitnikova, N.F. Morozov, Y.V. Petrov. Mathematics and Mechanics of Solids, 14, 72 (2009). DOI: 10.1177/1081286508092603
- [13] A.A. Gruzdkov, S.I. Krivosheev, Y.V. Petrov. Physics Solid State, 45, 886 (2003). DOI: 10.1134/1.1575328
- [14] G.I. Kanel, S.V. Razorenov, A.A. Bogatch, A.V. Utkin, V.E. Fortov, D.E. Grady. J. Appl. Phys., 20, 467 (1997). DOI: 10.1016/S0734-743X(97)87435-0
- [15] G.V. Garkushin, G.I. Kanel', S.V. Razorenov. Physics Solid State, 52, 2369 (2010). DOI: 10.1134/S1063783410110247
- [16] A.P. Zhilyaev, T.G. Langdon. Prog. in Mater. Sc., 53 (6), 893 (2008). DOI: 10.1016/j.pmatsci.2008.03.002
- [17] G.K. Williamson, R.E. Smallman. Phil. Mag., 1, 34 (1956).
 DOI: 10.1080/14786435608238074
- [18] H. Kolsky. Proceed. Phys. Society, 62, 676 (1949).

- [19] A.M. Bragov, A.K. Lomunov. Int. J. Impact. Engin., 16 (2), 321 (1995). DOI: 10.1016/0734-743X(95)93939-G
- [20] E.O. Hall. Proceed. Phys. Society, Section B, 64, 747 (1951).
- [21] N.J. Petch. Acta Crystallographica, 6, 96 (1953).DOI: 10.1107/S0365110X53000260
- [22] Yu.V. Petrov, Dokl. Akademii Nauk, 395 (5), 621 (2004).

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