OF MULTI-COMPONENT AI-Mg-Si ALLOYS DURING NANOINDENTATION TEST

Received: June 26, 2015

NANOINDENTATION TEST

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Abstract. This paper presents the research results of the effect of the difference in the sample and indenter temperature on the calculated characteristics of the mechanical properties of multi-component Al-Mg-Si alloys during nanoindentation test carried out on the AVT-1 alloy sample. The surface topography of the sample was studied using atomic force microscope and profilometer, after that 3 series of indentation experiments with different heating schemes were conducted, results are presented in the form of tables and illustrated with graphs, showing dependence of calculated characteristics of the material mechanical properties, determined using the nanoindentation platform software, from temperature.

1. Introduction

Modern high-tech industries such as biotechnology, nanoelectronics, energetics and mechanical engineering are placing increasing demands on the physical and mechanical properties of materials [1]. Nanoindentation holds leading position in the research of physical and mechanical properties of traditional and advanced materials in the micro- and nanoscale, representing today a broad class of techniques and methods of micro- and nanomechanical testing of materials, and related modes of contact atomic force microscopy. In a short time, they gave birth to a myriad of ideas and approaches to improve the creation of traditional and original materials, structures, coatings [2]. New physically justified models of the indentation process are currently developed [3-5] as well as the methods for extraction of real characteristics out of raw data for modern materials such as nanocomposites, polymers, ceramics, thin films, biomaterials, and others. Improved designs of nanoindentation platforms and expanding of their functionality create prerequisites for the study of temperature-dependent mechanical properties of metals and alloys with such devices [6-8 and others].

The aim of this study was nanoindentation of Al-Mg-Si alloys. The AVT-1 alloy sample was considered, which was subjected to conventional hardening heat treatment: quenching in water at 515-525 °C and aging treatment at 160 °C with 12-18 hours of exposure. In the aging process of such alloys (aircraft-grade aluminum) Mg and Si atoms form thermally stable compounds of magnesium silicide Mg₂Si (reinforcing phases [9]).

Due to its high corrosion resistance, large enough strength, high fatigue strength and toughness, the alloys of this system are widely used in the manufacture of propeller blades of helicopters, components for aviation, car frame and chassis, orthopedic implants and other constructions [10-12].

2. Experiment and results

All the experiments were held in the Resource Center for Collective Usage of Research and Education Center "Materials" of Don State Technical University [13]. Sample preparation was conducted before experiments: grinding using a paste based on chromium oxide (III). Immediately before nanoindentation the sample surface microgeometry was evaluated using profilometer Surftest SJ-210 (Mitutoyo, Japan) and atomic force microscope (AFM) Nanoeducator (NT-MDT, Russia) for determining the load which would be applied during the experiment (Figs. 1 and 2). The results are correlated: the average roughness ($R_a \approx 40$ nm) and the height of the roughness peaks ($R_t \approx 0.3~\mu m$) coincide. All indentations in this work were carried out with 20 mN load. For a given load, the depth of the indenter penetration into the sample is ~ 600 nm, which corresponds to the maximum height of surface roughness. The loading rate of the indenter is 0.45 mN/s, which made it possible to avoid undesirable noise in the experiment.

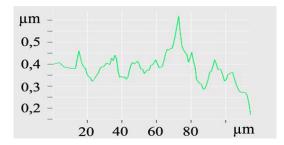


Fig. 1. Surface profile, acquired using AFM Nanoeducator.

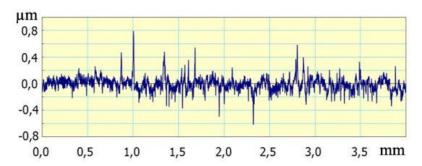


Fig. 2. Surface profile, acquired using profilometer Surftest SJ-210.

Nanoindentation was carried out using nanoindentation testing platform Nanotest 600 Platform 3 (Micro Materials, UK), equipped with a camera, in which the temperature was kept constant, with functional block «Nanotest», allowing to apply load in the range from 0.1 mN to 500 mN. In all experiments, a Berkovich indenter adapted for experiments at high temperature was used, with radius of curvature at the top ~ 100 nm. The nanoindentation analysis was performed using the Oliver – Pharr method.

«Hot stage» construction was used to perform heating: thermally insulating ceramic block with a sample holder. Between it and the pendulum, the thermal protection screen was mounted, preventing overheating of nanoindentation platform. The indenter was heated with special heating element. The sample was mounted on a sample holder using a high temperature silicone gasket maker Red RTV Silicone Gasket Maker (Doctor Chemical Corp., USA). It took 24 hours from glueing of the sample to the first experiment, that corresponds to the requirements of the gasket maker. During the experiment the temperature is controlled by temperature sensors Eurotherm 2216e (Eurotherm, UK). Before the experiments all the necessary nanoindentation platform calibrations were carried out using the calibration sample of fused silica.

Altogether three series of experiments were done, in each of which heating of the

indenter and the sample were carried out separately. For each pair of temperature values of the indenter and the sample between 8 and 12 indentations were performed at different spaced apart regions of the sample (situated not far from each other), with the average value of characteristics determined during indentation test was assumed as a result.

Series 1 - the temperature of the indenter was maintained constant at $60\,^{\circ}$ C, while the temperature of the sample was increased from experiment to experiment. The results of this series of experiments are presented in Table 1.

Series 2 – both the sample and the indenter were heated to the same temperature, which was increased from one experiment to another. The results of this series of experiments are presented in Table 2.

Series 3 - sample temperature was maintained constant at 60 °C, while the temperature of the indenter was increased from experiment to experiment. Results of this series of experiments are presented in Table 3.

Table 1. Results of series 1 of experiments.

№	Indenter temperature, °C	Sample temperature, °C	Results	
			Modulus of elasticity E, GPa	Hardness H, GPa
1	60	40	152.43 ± 6.10	1.94 ± 0.051
2	60	60	120.43 ± 5.59	1.95 ± 0.110
3	60	80	95.45 ± 5.13	2.15 ± 0.068
4	60	100	75.15 ± 3.63	2.59 ± 0.16
5	60	115	68.06 ± 2.02	3.23 ± 0.131

Table 2. Results of series 2 of experiments.

№	Indenter temperature, °C	Sample temperature, °C	Results	
			Modulus of elasticity E, GPa	Hardness H, GPa
1	40	40	121.38 ± 6.35	1.98 ± 0.2
2	60	60	120.43 ± 5.59	1.95 ± 0.110
3	80	80	130.63 ± 9.93	2.09 ± 0.2
4	100	100	149.23 ± 12.59	1.96 ± 0.22
5	115	115	150.43 ± 26.58	2.11 ± 0.084

Table 3. Results of series 3 of experiments.

№	Indenter temperature, °C	Sample temperature, °C	Results	
			Modulus of elasticity E, GPa	Hardness H, GPa
1	40	60	96.39 ± 5.34	2.28 ± 0.166
2	60	60	128.48 ± 3.42	2.035 ± 0.119
3	80	60	179.26 ± 11.79	1.9 ± 0.007
4	100	60	171.69 ± 3.74	1.7 ± 0.101
5	115	60	150.34 ± 8.57	1.34 ± 0.117

Figures 3 and 4 show graphs of modulus of elasticity and hardness dependence from temperature in three series of experiments.

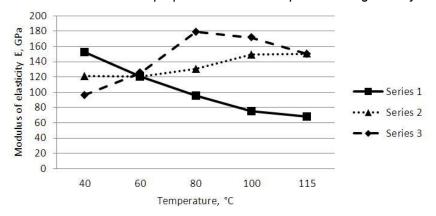


Fig. 3. Modulus of elasticity of AVT-1 alloy dependence from temperature acquired in three series of experiments.

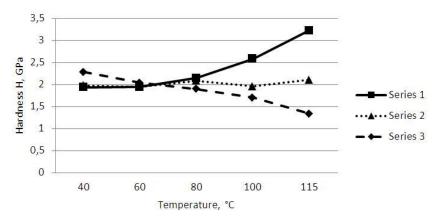


Fig. 4. Hardness of AVT-1 alloy dependence from temperature acquired in three series of experiments.

3. Conclusions

Investigation of the effect of temperature on the calculated mechanical properties of multi-component Al-Mg-Si alloys during nanoindentation test was conducted during the three series of experiments using different heating schemes with subsequent data interpretation by the standard software of the device according to the Oliver – Pharr method.

It is shown that the temperature difference and the heat flow between the indenter and the sample has a significant impact on the calculated mechanical characteristics of material, determined by the standard software of the device. Therefore, using the standard methods for determining the mechanical properties of materials requires one to perform simultaneous and uniform heating of both the indenter and the sample during nanoindentation test. Today rather small amount of devices for nanoindentation have ability to satisfy these requirements.

The study results demonstrate the dependence of the calculated hardness and modulus of elasticity of the AVT-1 alloy from temperature even at small difference in temperature of the indenter and the sample (heating to 115 °C) determined using the standard software of the device. It is shown that one must take into account the difference in temperature of the indenter and the sample during nanoindentation test for an adequate interpretation of the experimental results.

Acknowledgements

This work was partially supported by the Ministry of Education and Science of Russia and Russian Foundation for Basic Research (grants nos. 14-07-00343-a, 16-07-00958-a, 15-38-20790-mol_a_ved, 15-57-04084-Bel_mol_a).

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