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SURFACE ELECTRON BEAM ALLOYING LIGHT METALS USING TICN NANOPARTICLES

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Abstract
TiCN nanopowder deposited by an appropriate way on aluminum and aluminum alloys surface substrate is incorporated in the matrix using scanning electron beam. The samples are investigated by means of light microscopy, SEM, and EDX. The microhardness of all of them is determined. It is found out that uniform and dense coating with 7-30 µm thickness, well adherent to the substrate, is formed. In case of aluminum alloys, the microhardness of the modified zone is up to 2.4 times higher than the matrixes one. After the alloying pure aluminum with TiCN the measured microhardness is about 19 times higher than aluminum matrix.

Keywords: nanoparticles, electron beam, surface alloying, light metals.

INTRODUCTION

In the several recent years, nano-sized powders of refractory compounds are used for refining and modifying microstructure as well as for improving mechanical properties of metals and alloys. They are introduced in the melt by the appropriate way, homogenously distributed, wetted and absorbed by it.

The nanoparticles influence both microstructure formation and properties of the solidified metal. They are used in metal casting [1-4], welding [5-9] and surface alloying [10-15].

The aim of the present work is to introduce TiCN nanoparticles in aluminum and aluminum alloy substrate by scanning electron beam and to study the surface microstructure changes.

EXPOSITION

Materials and methods

We chose the refractory TiCN powder as a coating material component due to its excellent properties such as high melting point, extreme hardness, and high thermal conductivity [16]. The mean particle size is 40±5 nm and the C: N ratio is 1:1.

The aluminum used as a substrate is of technical purity – 99.5% Al and 0.5% other elements.

Two metallographic samples are wet ground to grinding paper 1200, cleaned with ethyl alcohol and dried. Then one of them (Nr 1) is smeared with a mixture of three drops $CHCl_3$, dissolved plastic rasping and TiCN nano powder for obtaining a film with concentration 0.03mg/mm^2 TiCN. The obtained film is uniform and well adherent to the aluminum substrate after drying. The as-prepared samples are subjected to an electron-beam treatment in installation type ESW300/60-15 ("Leybold – Heraeus") – *fig.1.* The electron beam scanning motion is circular rotating on the surface of the aluminum specimen which moves in a straight line. In this way a liquid pool is maintained for a long time, the melt is well stirred and the nanoparticles are mixed with the molten metal. As a result of the interaction of electron beam with TiCN nanoparticles film is created a coating on the surface of the aluminum specimen.

The parameters of treatment process are shown in *table 1.*

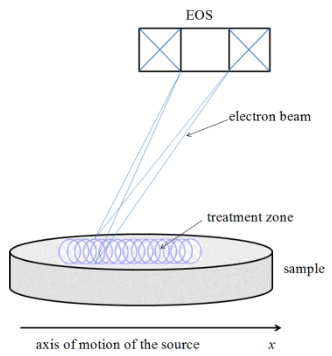


Fig. 1. *Scheme of electron beam treatment*

Table 1. *Parameters of electron beam treatment process of Al samples*

Nr of sample	I_b [mA]	v [cm/s]	f [kHz]	U [kV]	I_f [mA]	P [kW]
0	18	0.5	0.2	52	472	0.936
1	25	5	10	52	472	1.3

After electron beam treatment the metallographic samples are cut perpendicularly to the trace of the electron beam, wet ground on silicon carbide grinding paper Nr 1200 and 4000, and then electrolytic polished and etched by electropolishing device Lectropol (Struers) using the following electrolyte: 575 ml methyl alcohol, 25 ml $HClO_4$, 10 ml HNO_3. The observations are performed by means of metallographic microscope PolyvarMet with magnifications up to 1000x. The HV microhardness is measured by device MicroDuromat (Reichert-Jung) with a load of 20g, time for reaching the load 10s and holding time 10s. The SEM investigations are carried out with SEM/FIB LYRAI XMU, TESCAN, equipped with an EDX detector (Quantax 200, Brucker).

Metallographic samples of AlSi12Cu2NiMg alloy are wet ground to grinding paper 1200. Then one of them (Nr B1) is smeared with a mixture of three drops $CHCl_3$, plastic rasping and TiCN powder for obtaining a film with concentration 0.015 mg/mm^2 TiCN. The obtained film is uniform and well adherent to the substrate. The as-prepared samples are subjected to electron-beam treatment. The circular unwinding d=5,8 divisions. The electron beam treatment conditions are given in *table 2*.

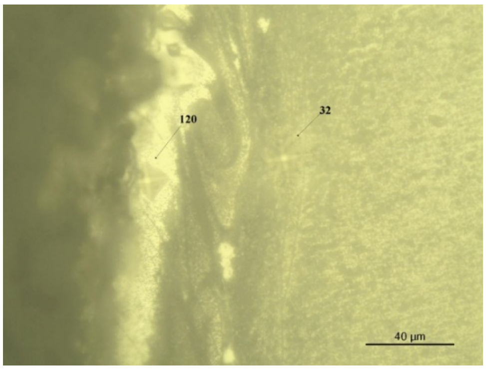
Table 2. *Parameters of electron beam treatment process of AlSi12Cu2NiMg samples*

Nr of sample	I_b [mA]	v [cm/s]	f [kHz]	U [kv]	I_f [mA]	P [kW]
B0	18	0.5	10	52	472	0.936
B1	25	3	1	52	472	1.3

After electron beam treatment the metallographic samples are wet ground on silicon carbide grinding paper 1200 and 4000 on the perpendicular of the coating cross-section. Then they are etched with 0.5% water solution of HF. The samples are investigated by means of light microscopy and the microhardness of all of them is determined.

Results and discussion

The optical and SEM images of aluminum samples are shown in *fig. 2*. There is a light layer well visible on the substrate surface. Its thickness reaches 30 µm. The layer's microhardness of the sample without nanoparticles on the surface is 4 times higher than the substrate's one. That means the electron beam treatment it selves modifies the surface structure and forms a hard layer on it. The layer of the sample smeared with nanoparticles and electron beam treated is 19 times higher than the substrate's one and 5 times higher than the layer without nanoparticles. Consequently, the nanoparticles strengthen the metal substrate surface and a very hard coating is formed. The proof of the nanoparticles penetration in the aluminum is the presence of Ti in the sample's surface which is registered by EDX detector in *fig. 3.* The TiCN nanoparticles are not only refractory and they do not melt in the molten metal, but they are very hard and commensurable with dislocations and represent obstacles for them. While the high hardness in the melted surface layer after irradiation with the electron beam can be explained by the residual stresses in it, the high hardness of the layer with incorporated nanoparticles can be explained by the reinforcement of the matrix with them.



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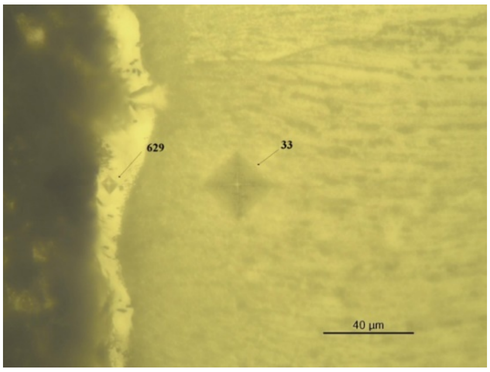

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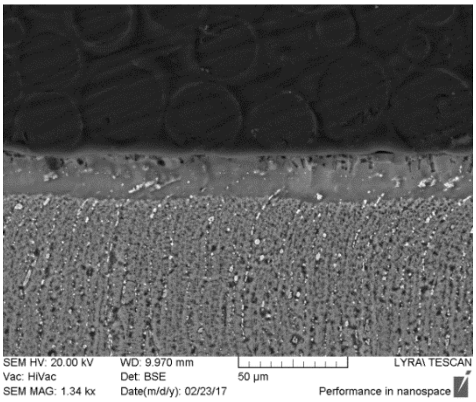

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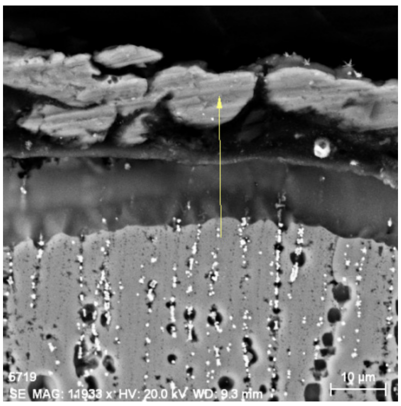
Fig. 2. *Optical and SEM microimages of aluminum samples: a) sample Nr 0 – irradiated, without nanoparticles; b) and c) sample Nr1 - smeared with nanoparticles and irradiated*



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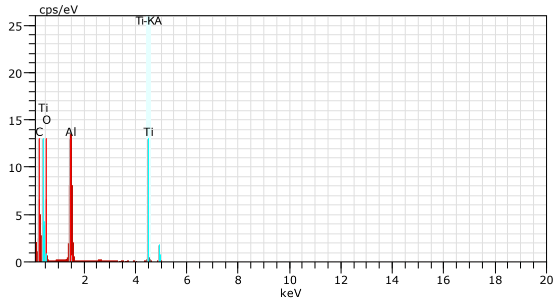

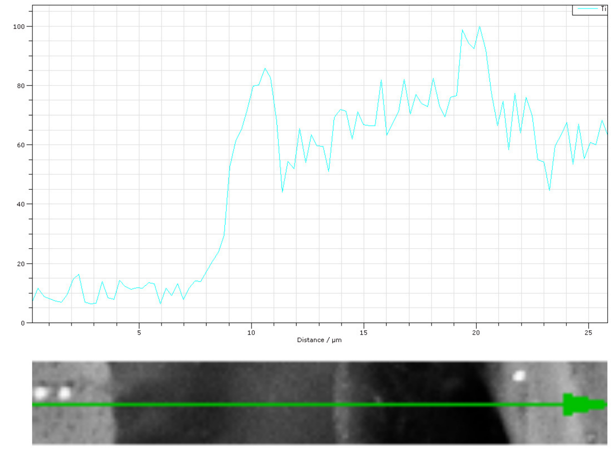

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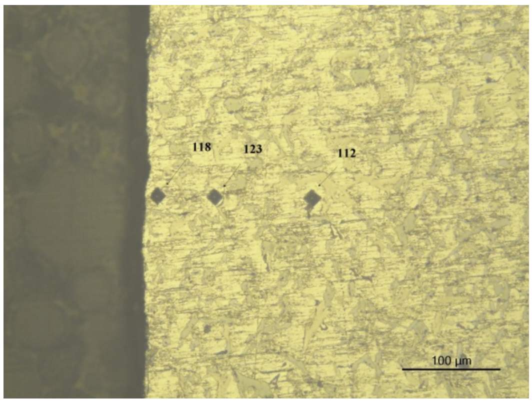
Fig. 3. *SEM image and EDS results for aluminum sample Nr1 - smeared with nanoparticles and irradiated*

The results from experiments with AlSi12Cu2NiMg samples are shown in *fig. 4*. No modified zone has been formed after electron beam treatment in the used conditions in a sample without nanoparticles. However, a thin modified zone with fine precipitations and thickness of about 20 µm has been formed at the sample's surface of the other sample. The microhardness of this coating is 2.4 times higher than the microhardness of the substrate while the microhardness of the sample without nanoparticles remains nearly unchangeable from the core to the surface – *fig. 4 a).*



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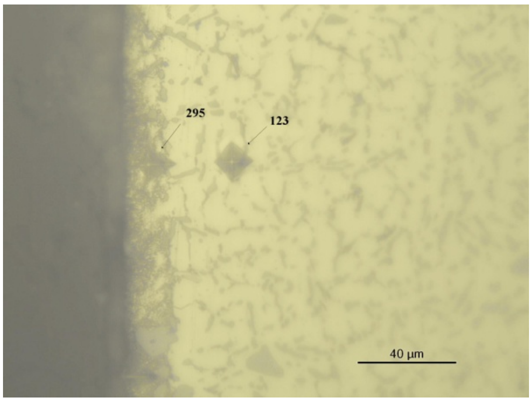

b

Fig. 4. *Optical microimages of AlSi12Cu2NiMg samples: a) sample Nr B0 – irradiated, without nanoparticles; b) sample Nr B1 - smeared with nanoparticles and irradiated*

We assume that the precipitates formation is initiated by the nanoparticles which are dispersed in the surface matrix because of grain and phase generating role of the nanoparticles during solidification. This assumption should be proved in the further investigation.

CONCLUSION

Following conclusions could be drawn from the carried out investigation:

1. The electron beam treatment modifies the surface structure of aluminum samples and forms layer which is 4 times harder than the substrate's one. This hardness is probably due to the residual stresses on the surface as a result of the irradiation.
2. A layer with 19 times higher microhardness than the core's one and 5 times higher than the layer without nanoparticles is formed on the sample with nanoparticles. That means the nanoparticles incorporation strengthen the metal substrate surface forming a very hard coating.
3. No modified zone has been formed after electron beam treatment in the used conditions in a sample of AlSi12Cu2NiMg without nanoparticles. The microhardness remains nearly unchangeable from the core to the surface.
4. Modified zone with fine precipitations and thickness of about 20 μm has been formed at the surface of AlSi12Cu2NiMg sample with nanoparticles. Its microhardness is 2.4 times higher than the microhardness of the substrate.
5. Assumption that the precipitates formation is initiated by the nanoparticles in the molten alloy because of their grain and phase generating role during solidification is made.

REFERENCE

[1] Krushenko G. G., Balashov B. A., Miller T.N. et al., New Technological Processes in Foundry. Materials of the Scientific and Technical Conference. Omsk, OmSTU, 1997, p. 20-22.

[2] R. Lazarova, R.H. Petrov, V. Gaydarova, A. Davidkov, A. Alexeev, M. Manchev, V. Manolov, Microstructure and mechanical properties of P265GH cast steel, after modification with TiCN particles, Materials and Design 32 (2011), pp. 2734–2741.

[3] Dimitrova, R., Kuzmanov, P., Lazarova, R. And Manolov, V., Investigation of nanopowders application in metal casting, 2013, Advanced Materials Research 629, pp. 284-291.

[4] R. Lazarova, N. Bojanova, R. Dimitrova, I. Panov and V. Manolov, Influence of Nanoparticles Introducing in the Melt of Aluminum Alloys on Castings Microstructure and Properties, International Journal of Metalcasting, 2016, Vol. 10, Issue 4, pp. 466-476.

[5] Troshkov A.S. Modification of the Weld Metal Structure by Nanodispersed Tungsten Carbides. Polzunovsky Almanac No. 2, 2009.

[6]. Drozdov, V.O. et al. An Experimental Study of Laser Welding of Metals Using Nanopowder Additives. Dokl. V All-Russian Conferences "Interaction of Highly Concentrated Energy Flows with Materials in Advanced Technologies and Medicine". Novosibisirsk, March 26-29, 2013, p. 97-101.

[7] Tashev P., T. Petrov, Y. Lukarsky, G. Stefanov. Technology for Nanosized Particle Insertion in Welding Seam in Welding Processes", Engineering Sciences, year L, 2013, № 3, Journal of Engineering Sciences Department, to the Bulgarian Academy of Sciences, p. 82-93.

[8] P. Tashev, R. Lazarova. Examination of Layer Overlaid with Addition of TiN Nano Particles, Mechanics Transport Communications, Vol. 11, Issue 1, 2013, XIII-1 – XIII-6, ISSN, p. 1312-3823.

[9] Tashev, P., R. Lazarova, M. Kandeva, R. Petrov and V. Manolov. Tungsten Inert Gas Weld Overlay using Nano-sized TiN Powder, Journal of the Balkan Tribological Association, Vol. 22, No 3, p. 2238-2254.

[10] Draper, C.W.; Poate, J.M. Laser surface alloying. *International Metal Review*, 30 (2), 1985, p. 85-108.

[11] Murzakov, M. A.; Chirikov, S. N.; Markushov, Y. V. Research on microstructure and wear resistance of coatings obtained by adding nanoparticles of refractory compounds in laser cladding. Journal of Physics: Conference Series 747, 2016, 012062, p. 1-4.

[12] Weipo, Xin; Deqiang, Wei. Study on the Microstructure Modification of 6061Aluminum by Electron Beam Surface Treatment. Sixth International Conference on Measuring Technology and Mechatronics Automation (2010), Changsha, China, Mar. 13, 2010 to Mar. 14, 2010, p. 665-668.

[13] Deqiang, Wei; Weipo Xin; Rong Wang. Study of electron-beam scanning on the surface of aluminum alloy with nano-sized Al_2O_3-Al powder. International Conference on Advanced Technology of Design and Manufacture (ATDM 2010), 2010, p. 357 – 360, Beijing, China.

[14] R. Lazarova, R. Dimitrova, St. Valkov, P. Petrov. Coatings Obtained by TiCN Nanopowder Deposition on Al Substrate and Subsequent Electron-Beam Treatment, Proceedings of 6th National conference with international participation "Metal science hydro- and aerodynamic, national security '2017", Sofia, in press.

[15] Song, R. G.; Zhang, K.; Chen, G.N. Electron beam surface treatment. Part II: microstructure evolution of stainless steel and aluminum alloy during electron beam rapid solidification. Vacuum 69, 2003, p. 517–520.

[16] Wenxia, Feng; Shouxin, Cui; Haiquan, Hu; Guiqing, Zhang; Zengtao, Lv. Electronic structure and elastic constants of TiC_xN_{1-x}, $Zr_xNb_{1-x}C$ and HfC_xN_{1-x} alloys: A first-principles study. *Physica* B 406, 2011, p. 3631–3635

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